## RENEWAL APPLICATIONS FOR VPDES PERMIT VA0090981

### TO DISCHARGE TREATED WASTEWATER AND STORM WATER ASSOCIATED WITH INDUSTRIAL ACTIVITIES

ILUKA RESOURCES INC
VIRGINIA OPERATIONS
MINERAL SEPARATION PLANT
STONY CREEK, VA

July 2007

JUL 2 4 2007

EPA I.D. NUMBER (copy from Item 1 of Form 1)

Please print or type in the unshaded areas only.

VA0090981

Form Approved PRO OMB No. 2040-0086. Approval expires 3-31-98.

FORM 2C **\$EPA** 

U.S. ENVIRONMENTAL PROTECTION AGENCY APPLICATION FOR PERMIT TO DISCHARGE WASTEWATER EXISTING MANUFACTURING, COMMERCIAL, MINING AND SILVICULTURE OPERATIONS Consolidated Permits Program

| For each outfall, list the I<br>A. OUTFALL NUMBER |         | B. LATITUDE |         |         | LONGITUDE |         |   |
|---|---------|-------------|---------|---------|-----------|---------|---|
| (list)  | 1. DEG. | 2. MIN.     | 3. SEC. | 1. DEG. | 2. MIN.   | 3. SEC. | D. RECEIVING WATER (name)               |
| 001   | 36      | 58          | 10.41   | 77      | 24        | 11.75   | GALLEY SWAMP                            |
|   |         |             |         |         |           |         | see Appendix 1, Fig. 1 for location map |
|   |         |             |         |         |           |         |   |
|   |         |             |         |         |           |         |   |

- A. Attach a line drawing showing the water flow through the facility. Indicate sources of intake water, operations contributing wastewater to the effluent, and treatment units labeled to correspond to the more detailed descriptions in Item B. Construct a water balance on the line drawing by showing average flows between intakes, operations, treatment units, and outfalls. If a water balance cannot be determined (e.g., for certain mining activities), provide a pictorial description of the nature and amount of any sources of water and any collection or treatment measures. Please See APPENDIX 1, FIGURE 3.

  B. For each outfall, provide a description of: (1) All operations contributing wastewater to the effluent, including process wastewater, sanitary wastewater, cooling water, and storm water runoff; (2) The average flow contributed by each operation; and (3) The treatment received by the wastewater. Continue on additional sheets if

| 1. OUT-            | 2. OPERATION(S) CO   | ONTRIBUTING FLOW                | 3. TREATME                 | NT                    |                  |
|--------------------|--|---------------------------------|----------------------------|-----------------------|------------------|
| FALL<br>NO. (list) | The state of the s | b. AVERAGE FLOW (include units) | a. DESCRIPTION             | b. LIST CO<br>TABL    | DDES FROM        |
| 001                | ZIRCON FINISHING PLANT (ZFP)<br>EFFLUENT TREATMENT PLANT (ETP)   | 29.4 gpm                        | See Appendix 2, Document 1 | 2-C, 2-K,<br>4-A, 4-C | 5-L, 5-Q,<br>5-U |
|                    |  |                                 |                            |                       |                  |
|                    |  |                                 |                            |                       |                  |
|                    |  |                                 |                            |                       |                  |
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|                    |  |                                 |                            |                       |                  |
|                    |  |                                 |                            | :e:C                  |                  |
|                    |  |                                 |                            |                       |                  |
|                    |  |                                 |                            |                       |                  |
|                    |  |                                 |                            | *                     |                  |
|                    |  |                                 |                            |                       |                  |
|                    |  |                                 |                            |                       |                  |
|                    |  |                                 |                            |                       |                  |
|                    |  |                                 |                            |                       |                  |
|                    |  |                                 |                            |                       |                  |
|                    |  |                                 |                            |                       |                  |
|                    |  |                                 |                            |                       |                  |
|                    |  |                                 |                            |                       |                  |

OFFICIAL USE ONLY (effluent guidelines sub-categories)

CONTINUED FROM THE FRONT C. Except for storm runoff, leaks, or spills, are any of the discharges described in Items II-A or B intermittent or seasonal? YES (complete the following table) NO (go to Section III) 3. FREQUENCY 4. FLOW a. DAYS PER B. TOTAL VOLUME 2 OPERATION(s) b. MONTHS PER YEAR a. FLOW RATE (in mgd) WEEK 1. OUTFALL NUMBER (list) CONTRIBUTING FLOW (specify 1. LONG TERM 2. MAXIMUM AVERAGE DAILY 2. MAXIMUM C DURATION 1. LONG TERM (list) average) (specify average (in days) AVERAGE DAILY Zircon Finishing Plant Effluent (ZFP) Treatment Plant (ETP) 0.63 .04234 .05040 42,444 gpd 50,400 gpd average average III. PRODUCTION A. Does an effluent guideline limitation promulgated by EPA under Section 304 of the Clean Water Act apply to your facility? YES (complete Item III-B) NO (go to Section IV) B. Are the limitations in the applicable effluent guideline expressed in terms of production (or other measure of operation)? YES (complete Item III-C) NO (go to Section IV) C. If you answered "yes" to Item III-B, list the quantity which represents an actual measurement of your level of production, expressed in the terms and units used in the applicable effluent guideline, and indicate the affected outfalls. 1. AVERAGE DAILY PRODUCTION 2. AFFECTED OUTFALLS c. OPERATION, PRODUCT, MATERIAL, ETC. a. QUANTITY PER DAY b. UNITS OF MEASURE (list outfall numbers) (specify) na na IV. IMPROVEMENTS A. Are you now required by any Federal, State or local authority to meet any implementation schedule for the construction, upgrading or operations of wastewater treatment equipment or practices or any other environmental programs which may affect the discharges described in this application? This includes, but is not limited to, permit conditions, administrative or enforcement orders, enforcement compliance schedule letters, stipulations, court orders, and grant or loan conditions. YES (complete the following table) NO (go to Item IV-B) 1. IDENTIFICATION OF CONDITION. 2. AFFECTED OUTFALLS 4. FINAL COMPLIANCE DATE 3. BRIEF DESCRIPTION OF PROJECT AGREEMENT, ETC. a. NO b. SOURCE OF DISCHARGE a. REQUIRED b. PROJECTED na na na na B. OPTIONAL: You may attach additional sheets describing any additional water pollution control programs (or other environmental projects which may affect your discharges) you now have underway or which you plan. Indicate whether each program is now underway or planned, and indicate your actual or planned schedules for construction. MARK "X" IF DESCRIPTION OF ADDITIONAL CONTROL PROGRAMS IS ATTACHED

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| V. INTAKE AND EFFLUENT CHARACTE   | RISTICS   |  |  |
|---|---|--|--|
| NOTE: Tables V-A, V-B, and  | V-C are included on separate sheets numbe   | outfall – Annotate the outfall number in the ered V-1 through V-9. |  |
| <ul> <li>Use the space below to list any of the<br/>from any outfall. For every pollutant yo</li> </ul> | pollutants listed in Table 2c-3 of the instru-<br>ou list, briefly describe the reasons you belie   | ctions, which you know or have reason to b                         | elieve is discharged or may be discharged data in your possession. |
| 1. POLLUTANT  | 2. SOURCE   | 1. POLLUTANT   | 2. SOURCE  |
| Zirconium   | Zircon sand product. Zirconium and compounds containing Zirconium are believed absent due to the insolubility of zircon under the conditions it is subjected to in the MSP and ZFP.   |  |  |
| VI. POTENTIAL DISCHARGES NOT COV  | Iron is present as a naturally occuring oxide coating on zircon grains. The purpose of the ZFP is to remove this coating. Most of this iron is precipicated out with the gypsum in the ETP. Samples from outfall 001 have been analyized for iron. Results can be found in this form and in Appendix 3 as Item 2. |  |  |
|   |   | ou currently use or manufacture as an interr                       | modiate or final product or hungarity                              |
| YES (list all such pollutants   | below)  | NO (go to Item VI-B)   | nodate of final product of byproduct?                              |
|   |   |  |  |
|   |   |  |  |

#### CONTINUED FROM THE FRONT

| VII. BIOLOGICAL TOXICITY TESTING DAT  | A   |  |  |
|---|---|--|--|
| Do you have any knowledge or reason to be relation to your discharge within the last 3 years. | lieve that any biological test for acute or chronic tox   | icity has been made on any of your                                     | discharges or on a receiving water in  |
| YES (identify the test(s) and de  |   | NO (go to Section VIII)  |  |
| One Whole Effluent Toxicity<br>Virginia Department of Envir                                   | (WET) sample was taken from outfa<br>onmental Quality's (DEQ) Virginia<br>les were submitted to Coastal Bio   | ll 001 on July 26, 2006  | mination Drogram (UDDEC)   |
| VIII. CONTRACT ANALYSIS INFORMATION   |   | gr. r  |  |
|   | performed by a contract laboratory or consulting firr   | -2   |  |
|   | d telephone number of, and pollutants analyzed by,  | NO (go to Section IX)  |  |
| A. NAME   | B. ADDRESS  | C. TELEPHONE<br>(area code & no.)                                      | D. POLLUTANTS ANALYZED (list)  |
| Primary Laboratories  | 7423 Lee Davis Road<br>Mechanicsville, VA 23111   | 804-559-9004   | See Appendix 3, Item 2   |
|   |   |  |  |
| IV CERTIFICATION  |   |  |  |
| directly responsible for gathering the information  | ent and all attachments were prepared under my di<br>aluate the information submitted. Based on my inq<br>ition, the information submitted is, to the best of my<br>information, including the possibility of fine and impr | uiry of the person or persons who<br>knowledge and belief true accurat | manage the system or those persons<br>e, and complete. I am aware that there |

PLEASE PRINT OR TYPE IN THE UNSHADED AREAS ONLY. You may report some or all of this information EPA.I.D. N on separate sheets (use the same format) instead of completing these pages.

SEE INSTRUCTIONS.

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b. NO. OF ANALYSES OUTFALL NO. (2) MASS 001 4. INTAKE a. LONG TERM AVERAGE VALUE CONCENTRATION VALUE VALUE VALUE b. MASS STANDARD UNITS 3. UNITS (specify if blank) S Ö a. CONCENTRATION PART A -You must provide the results of at least one analysis for every pollutant in this table. Complete one table for each outfall. See instructions for additional details. mg/L mg/L mg/L mg/L mg/I d. NO. OF ANALYSES 14  $\vdash$  $\vdash$ 1 4 (2) MASS c. LONG TERM AVRG. VALUE (if available) mdb na na (1) CONCENTRATION 29.4 0.115 19.06 VALUE VALUE 2. EFFLUENT b. MAXIMUM 30 DAY VALUE (2) MASS MAXIMUM 8.96 V. INTAKE AND EFFLUENT CHARACTERISTICS (continued from page 3 of Form 2-C) (if available) na na na CONCENTRATION MINIMUM 6.02 VALUE VALUE VALUE MAXIMUM 7.06 a. MAXIMUM DAILY VALUE (2) MASS gpm 35.5 7.3 (1) CONCENTRATION 35 22.1 MINIMUM 6.15 <3.0 52.8 0.27 5.38 VALUE VALUE VALUE c. Total Organic Carbon (70C) a. Biochemical Oxygen b. Chemical OxygenDemand (COD) 1. POLLUTANT d. Total Suspended Solids (755) e. Ammonia (as N) Demand (BOD) h. Temperature g. Temperature (winter) f. Flow i. pH

Mark "X" in column 2-a for each pollutant you know or have reason to believe is present. Mark "X" in column 2-b for each pollutant you believe to be absent. If you mark column 2a for any pollutant with a limitations guideline, you must provide the results of at least one analysis for that pollutant. For other pollutants for which you mark column 2a, you must provide the results of at least one analysis for that pollutants for which you mark column 2a, you must provide quantitative data or an explanation of their presence in your discharge. Complete one table for each outfall. See the instructions for additional details and requirements. PART B -

|                                |         | 2. MARK "X" |                        |           | 3.                                  | 3. EFFLUENT | 3. EFFLUENT 4. UNITS  |            |                       | 4. UNITS         | S | 5. INT                         | 5. INTAKE (optional) | (7)                   |
|--------------------------------|---------|-------------|------------------------|-----------|-------------------------------------|-------------|---|------------|-----------------------|------------------|---|--------------------------------|----------------------|-----------------------|
| AND CAS NO                     |         | a.          | a. MAXIMUM DAILY VALUE | ILY VALUE | b. MAXIMUM 30 DAY<br>(if available) | DAY VALUE   | b. MAXIMUM 30 DAY VALUE c. LONG TERM AVRG. VALUE (if available) | VRG. VALUE |                       |                  |   | a. LONG TERM AVERAGE<br>VALUE  | AVERAGE              |                       |
| (if available)                 | PRESENT | ABSENT      | (1)<br>CONCENTRATION   | (2) MASS  | (1)<br>CONCENTRATION                | (2) MASS    | (1)<br>CONCENTRATION  | (2) MASS   | d. NO. OF<br>ANALYSES | a. CONCENTRATION |   | b. MASS CONCENTRATION (2) MASS | (2) MASS             | b. NO. OF<br>ANALYSES |
| a. Bromide<br>(24959-67-9)     |         | ×           |                        |           |                                     |             |   |            |                       |                  |   |                                |                      |                       |
| b. Chlorine, Total<br>Residual |         | ×           |                        |           |                                     |             |   |            |                       |                  |   |                                |                      |                       |
| c. Color                       |         | ×           |                        |           |                                     |             |   |            |                       |                  |   |                                |                      |                       |
| d. Fecal Coliform              |         |             | 73                     |           |                                     |             |   |            | Н                     | MPN/100mL        |   |                                |                      |                       |
| e. Fluoride<br>(16984-48-8)    |         | ×           |                        |           |                                     |             |   |            |                       |                  |   |                                |                      |                       |
| f. Nitrate-Nitrite (as N)      |         | ×           |                        |           |                                     |             |   |            |                       | mg/L             |   |                                |                      |                       |

CONTINUE ON REVERSE

ITEM V-B CONTINUED FROM FRONT

| TIEM V-B CONTINUED FROM FRONT                       | INCED FRO    | INDR IND |                        |           |  |             |   |            |                       |                  |         |                               |                       |
|---|--------------|----------|------------------------|-----------|--|-------------|---|------------|-----------------------|------------------|---------|-------------------------------|-----------------------|
| 1 POLLITANT   | V VANAIVI .2 | × ×      |                        |           | 3.                                     | 3. EFFLUENI |   |            |                       | 4. UNITS         | TS      | 5. INTAKE                     | 5. INTAKE (optional)  |
| AND   | Ġ.           | Ď.       | a. MAXIMUM DAILY VALUE | JLY VALUE | b. MAXIMUM 30 DAY VALUE (if available) | DAY VALUE   | c. LONG TERM AVRG. VALUE (if available) | VRG. VALUE |                       |                  |         | a. LONG TERM<br>AVERAGE VALUE |                       |
| (if available)                                      | PRESENT      | BELIEVED | (1)<br>CONCENTRATION   | (2) MASS  | (1)<br>CONCENTRATION                   | (2) MASS    | (1)<br>CONCENTRATION                    | (2) MASS   | d. NO. OF<br>ANALYSES | a. CONCENTRATION | b. MASS | (1)<br>CONCENTRATION          | b. NO. OF<br>ANALYSES |
| g. Nitrogen,<br>Total Organic (as<br>N)             |              | ×        |                        |           |  |             |   |            |                       |                  |         |                               |                       |
| h. Oil and<br>Grease                                |              | ×        |                        |           |  |             |   |            |                       |                  |         |                               |                       |
| i. Phosphorus<br>(as P), Total<br>(7723-14-0)       |              | ×        |                        |           |  |             |   |            |                       |                  |         |                               |                       |
| j. Radioactivity                                    |              |          |                        |           |  |             |   |            |                       |                  |         |                               |                       |
| (1) Alpha, Total                                    | ×            |          | 6.0 ± 0.7              |           |  |             |   |            | П                     | pci/L            |         |                               |                       |
| (2) Beta, Total                                     | ×            |          | 21 ± 3                 |           |  |             |   |            | П                     | pci/L            |         |                               |                       |
| (3) Radium,<br>Total                                |              | ×        |                        |           |  |             |   |            |                       |                  |         |                               |                       |
| (4) Radium 226,<br>Total                            |              | ×        |                        |           |  |             |   |            |                       |                  |         |                               |                       |
| k. Sulfate<br>(as SO <sub>1</sub> )<br>(14808-79-8) |              | ×        |                        |           |  |             |   |            |                       |                  |         |                               |                       |
| I. Sulfide (as S)                                   |              | ×        |                        |           |  |             |   |            |                       |                  |         |                               |                       |
| m. Sulfite<br>(as SO <sub>1</sub> )<br>(14265-45-3) |              | ×        |                        |           |  |             |   |            |                       |                  |         |                               |                       |
| n. Surfactants                                      |              | ×        |                        |           |  |             |   |            |                       |                  |         |                               |                       |
| o. Aluminum.<br>Total<br>(7429-90-5)                |              | ×        |                        |           |  |             |   |            |                       |                  |         |                               |                       |
| p. Barium, Total<br>(7440-39-3)                     |              | ×        |                        |           |  |             |   |            |                       |                  |         |                               |                       |
| q. Boron, Total<br>(7440-42-8)                      |              | ×        |                        |           |  |             |   |            |                       |                  |         |                               |                       |
| r. Cobalt, Total<br>(7440-48-4)                     |              | ×        |                        |           |  |             |   |            |                       |                  |         |                               |                       |
| s. Iron, Total<br>(7439-89-6)                       | ×            |          | 0.364                  |           |  |             |   |            | 2                     | mg/L             |         |                               |                       |
| t. Magnesium,<br>Total<br>(7439-95-4)               |              | ×        |                        |           |  |             |   |            |                       |                  |         |                               |                       |
| u. Molybdenum,<br>Total<br>(7439-98-7)              |              | ×        |                        |           |  |             |   |            |                       |                  |         |                               |                       |
| v. Manganese,<br>Total<br>(7439-96-5)               |              | ×        |                        |           |  |             |   |            |                       |                  |         |                               |                       |
| w. Tin. Total<br>(7440-31-5)                        |              | ×        |                        |           |  |             |   |            |                       |                  |         |                               |                       |
| x. Titanium,<br>Total<br>(7440-32-6)                |              | ×        | ·                      |           |  |             | 5                                       |            |                       |                  |         |                               |                       |
| EPA Form 3510-2C (8-90)                             | 2C (8-90)    |          |                        |           |  |             | PAGE V-2                                |            |                       |                  |         | CONT                          | CONTINUE ON PAGE V-3  |

OUTFALL NUMBER EPA I.D. NUMBER (copy from Item 1 of Form 1)

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CONTINUED FROM PAGE 3 OF FORM 2-C

PART C - If you are a primary industry and this outfall contains process wastewater, refer to Table 2c-2 in the instructions to determine which of the GC/MS fractions you must test for. Mark "X" in column 2-a (secondary industries, nonprocess wastewater outfalls, and nonrequired GC/MS fractions that apply to your industry and for ALL toxic metals, cyanides, and total phenols. If you are not required to mark column 2-c for each pollutant you believe is absent. If you mark column 2 for any pollutant, you must provide the results of at least one analysis for that pollutant. If you mark column 2b for any pollutant, you must provide the results of at least one analysis for that pollutant if you mark column 2b for acrolein, acrylonitrile, 24 dinitrophenol, you must provide the results of at least one analysis for each of these pollutants which you know or have reason to believe that you discharge in concentrations of 100 ppb or greater. If you mark column 2b for acrolein, acrylonitrile, 24 dinitrophenol, you which you wank column 2b for acrolein, acrylonitrile, 24 dinitrophenol, you which you wank column 2b for acrolein, acrylonitrile, 24 dinitrophenol, you which you wank column 2b for acrolein, acrylonitrile, 24 dinitrophenol, you which you wank column 2b you must periter submit at least one analysis for being discharge in concentrations of 100 ppb or greater. Otherwise, for pollutants for which you wank column 2b you wust either submit at least one analysis for beingly describe the reasons the pollutant is expected to be discharged. Note that there are 7 pages to this part, please review each carefully. Complete one table (all 7 pages) for each outfall. additional details and requirements.

ANALYSES 5. INTAKE (optional) (2) MASS a. LONG TERM AVERAGE VALUE (1) CONCENTRATION b. MASS 4. UNITS a. CONCENTRATION CONCENTRATION (2) MASS ANALYSES c. LONG TERM AVRG. VALUE (if available) b. MAXIMUM 30 DAY VALUE (if available) (2) MASS 3. EFFLUENT (1) CONCENTRATION a. MAXIMUM DAILY VALUE (2) MASS (1) CONCENTRATION BELIEVED BELIEVED PRESENT ABSENT METALS, CYANIDE, AND TOTAL PHENOLS 2. MARK "X" a. TESTING REQUIRED 1. POLLUTANT CAS NUMBER (if available)

mg/L mg/L mg/L mg/L mg/L mg/L mg/L mg/L mg/L /Su /Bu /Bu /Bm -Н Н  $\vdash$  $\vdash$ m -N N N  $\vdash$ <0.100 <0.050 <0.010 <0.020 <0.020 <0.0002 <0.005 <0.020 <0.010 <0.020 0.021 0.044 0.064 × × × × × × × × × × × × × × × 1M. Antimony, Total (7440-36-0) 4M. Cadmium, Total (7440-43-9) 3M. Beryllium, Total (7440-41-7) 11M. Silver, Total (7440-22-4) 8M. Mercury, Total (7439-97-6) Total (7440-28-0) 2M. Arsenic, Total (7440-38-2) 13M. Zinc, Total (7440-66-6) 5M. Chromium, Total (7440-47-3) 6M. Copper, Total (7440-50-8) 10M. Selenium, Total (7782-49-2) 7M. Lead, Total (7439-92-1) 9M. Nickel, Total (7440-02-0) 14M. Cyanide, Total (57-12-5) 15M. Phenols, Total 12M. Thallium, DIOXIN

× 2,3,7,8-Tetra-chlorodibenzo-P-Dioxin (1764-01-6)

EPA Form 3510-2C (8-90)

Below detection limit of 10 µg/L.

DESCRIBE RESULTS

PAGE V-3

CONTINUE ON REVERSE

CONTINUED FROM THE FRONT

| 2.   |                           | 2. MARK "X"               | 2                  |   | 3 FFFI LIFNT                              |                    |                       | OTIMIT A   | 0       | TANTAN D      | 4        |           |
|--|---------------------------|---------------------------|--------------------|---|---|--------------------|-----------------------|------------|---------|---------------|----------|-----------|
|  |                           |                           |                    |   | b. MAXIMUM 30 DAY VALUE                   | c. LONG TERM AVRG. |                       | 180        | 0       | a. LONG TER   | ONG TERM |           |
| CAS NUMBER<br>(if available)                     | a.<br>TESTING<br>REQUIRED | b.<br>BELIEVED<br>PRESENT | BELIEVED<br>ABSENT | a. MAXIMUM DAILY VALUE (1) CONCENTRATION (2) MASS | (if available) (1) CONCENTRATION (2) MASS | U.                 | d. NO. OF<br>ANALYSES | a. CONCEN- | b. MASS | AVERAGE VALUE | 0        | b. NO. OF |
| GC/MS FRACTION - VOLATILE COMPOUNDS              | - VOLATIL                 | LE COMPOL                 | SONO               | 1   |   | 00000              |                       |            |         |               | (Z) MASS |           |
| 1V. Accrolein<br>(107-02-8)                      | ×                         |                           |                    | < 5   |   |                    | rH                    | hg/L       |         |               |          |           |
| 2V. Acrylonitrile<br>(107-13-1)                  | ×                         |                           |                    | < 55  |   |                    | 171                   | hg/L       |         |               |          |           |
| 3V. Benzene<br>(71-43-2)                         | ×                         |                           |                    | < 5   |   |                    | П                     | ng/L       |         |               |          |           |
| 4V. Bis (Chloro-<br>methyl) Ether<br>(542-88-1)  |                           |                           | ×                  |   |   |                    |                       |            |         |               |          |           |
| 5V. Bromoform<br>(75-25-2)                       | ×                         |                           |                    | < 5   |   |                    | н                     | ng/L       |         |               |          |           |
| 6V. Carbon<br>Tetrachloride<br>(56-23-5)         | ×                         |                           |                    | 50  |   |                    | н                     | ng/L       |         |               |          |           |
| 7V. Chlorobenzene<br>(108-90-7)                  | ×                         |                           |                    | < 5   |   |                    | П                     | ng/L       |         |               |          |           |
| 8V. Chlorodi-<br>bromomethane<br>(124-48-1)      | ×                         |                           |                    | <5  |   |                    | Н                     | hg/L       |         |               |          |           |
| 9V. Chloroethane (75-00-3)                       |                           |                           | ×                  |   |   |                    |                       |            |         |               |          |           |
| 10V. 2-Chloro-<br>ethylvinyl Ether<br>(110-75-8) |                           |                           | ×                  |   |   |                    |                       |            |         |               |          |           |
| 11V. Chloroform<br>(67-66-3)                     | ×                         |                           |                    | S >   |   |                    | Н                     | hg/L       |         |               |          |           |
| 12V. Dichloro-<br>bromomethane<br>(75-27-4)      | ×                         |                           |                    | <.5   |   |                    | н                     | ng/L       |         |               |          |           |
| 13V. Dichloro-<br>difluoromethane<br>(75-71-8)   |                           |                           | ×                  |   |   |                    |                       |            |         |               |          |           |
| 14V. 1,1-Dichloro-<br>ethane (75-34-3)           | ×                         |                           |                    | < 5   |   |                    | П                     | ng/L       |         |               |          |           |
| 15V. 1,2-Dichloro-<br>ethane (107-06-2)          | ×                         |                           |                    | < 5   |   |                    | П                     | hg/L       |         |               |          |           |
| 16V. 1,1-Dichloro-<br>ethylene (75-35-4)         | ×                         |                           |                    | < 5   |   |                    | н                     | ng/L       |         |               |          |           |
| 17V. 1,2-Dichloro-<br>propane (78-87-5)          | ×                         |                           |                    | <5  |   |                    | П                     | ng/L       |         |               |          |           |
| 18V. 1,3-Dichloro-<br>propylene<br>(542-75-6)    | ×                         |                           |                    | < 5   |   |                    | н                     | T/Bn       |         |               |          |           |
| 19V. Ethylbenzene<br>(100-41-4)                  | ×                         |                           |                    | <5  |   |                    | П                     | ng/L       |         |               |          |           |
| 20V. Methyl<br>Bromide (74-83-9)                 | ×                         |                           |                    | <5  |   |                    | П                     | ng/L       |         |               |          |           |
| 21V. Methyl<br>Chloride (74-87-3)                |                           |                           | ×                  |   |   |                    |                       |            |         |               |          |           |
| 1  | DOM: COURSE               |                           |                    |   |   |                    |                       |            |         |               |          |           |

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|   | 2          | 2. MARK "X" |             |                               | 3 FFEI I I ENT            |                        |                       | 1                | OF      | TO A THAT IS                  | W            |                       |
|---|------------|-------------|-------------|-------------------------------|---------------------------|------------------------|-----------------------|------------------|---------|-------------------------------|--------------|-----------------------|
| 1. POLLUTANT                                      |            |             |             |                               | b. MAXIMIM 30 DAY VALUE   |                        |                       | 4. UNII S        | 0       | 5. IN IAKE (optional)         | = (optional) |                       |
|   | 9,         | ,<br>i      | o l         | a. MAXIMUM DAILY VALUE        | -                         | VALUE (if available)   |                       |                  |         | a. LONG TERM<br>AVERAGE VALUE | N.           |                       |
| (if available)                                    | REQUIRED   | PRESENT     | ABSENT      | (1)<br>CONCENTRATION (2) MASS | SS CONCENTRATION (2) MASS | CONCENTRATION (2) MASS | d. NO. OF<br>ANALYSES | a. CONCENTRATION | b. MASS | (1)<br>CONCENTRATION (2       | ASS          | b. NO. OF<br>ANALYSES |
| GC/MS FRACTION - VOLATILE COMPOUNDS (continued)   | - VOLATILI | E COMPOL    | JNDS (cont. | inued)                        |                           |                        |                       |                  |         | 4                             |              |                       |
| 22V. Methylene<br>Chloride (75-09-2)              |            |             | ×           |                               |                           |                        |                       |                  |         |                               |              |                       |
| 23V. 1,1,2,2-<br>Tetrachloroethane<br>(79-34-5)   | ×          |             |             | <.5                           |                           |                        | Н                     | ng/L             |         |                               |              |                       |
| 24V. Tetrachloro-<br>ethylene (127-18-4)          | ×          |             |             | ru ^                          |                           |                        | п                     | hg/L             |         |                               |              |                       |
| 25V. Toluene<br>(108-88-3)                        | ×          |             |             | N >                           |                           |                        | d                     | hg/L             |         |                               |              |                       |
| 26V. 1,2-Trans-<br>Dichloroethylene<br>(156-60-5) | ×          |             |             | 150                           |                           |                        | н                     | ng/L             |         |                               |              |                       |
| 27V. 1,1,1-Trichloro-<br>ethane (71-55-6)         |            |             | ×           |                               |                           |                        |                       |                  |         |                               |              |                       |
| 28V. 1,1,2-Trichloro-<br>ethane (79-00-5)         | ×          |             |             | < 5                           |                           |                        | Н                     | µg/L             |         |                               |              |                       |
| 29V Trichloro-<br>ethylene (79-01-6)              | ×          |             |             | < 55                          |                           |                        | н                     | hg/L             |         |                               |              |                       |
| 30V. Trichloro-<br>fluoromethane<br>(75-69-4)     |            |             | ×           |                               |                           |                        |                       |                  |         |                               |              |                       |
| 31V. Vinyl Chloride<br>(75-01-4)                  | ×          |             |             | rv.                           |                           |                        | П                     | µg/L             |         |                               |              |                       |
| GC/MS FRACTION - ACID COMPOUNDS                   | - ACID CON | MPOUNDS     |             |                               |                           |                        |                       |                  |         |                               |              |                       |
| 1A, 2-Chlorophenol<br>(95-57-8)                   | ×          |             |             | <10                           |                           |                        | н                     | ng/L             |         |                               |              |                       |
| 2A, 2,4-Dichloro-<br>phenol (120-83-2)            | ×          |             |             | <10                           |                           |                        | П                     | hg/L             |         |                               |              |                       |
| 3A. 2,4-Dimethyl-<br>phenol (105-67-9)            | ×          |             |             | < 10                          |                           |                        | п                     | hg/L             |         |                               |              |                       |
| 4A. 4,6-Dinitro-O-<br>Cresol (534-52-1)           |            |             | ×           |                               |                           |                        |                       |                  |         |                               |              |                       |
| 5A. 2,4-Dinitro-<br>phenol (51-28-5)              |            |             | ×           |                               |                           |                        |                       |                  |         |                               |              |                       |
| 6A. 2-Nitrophenol<br>(88-75-5)                    |            |             | ×           |                               |                           |                        |                       |                  |         |                               |              |                       |
| 7A, 4-Nitrophenol (100-02-7)                      |            |             | ×           |                               |                           |                        |                       |                  |         |                               |              |                       |
| 8A. P-Chloro-M-<br>Cresol (59-50-7)               |            |             | ×           |                               |                           |                        |                       |                  |         |                               |              |                       |
| 9A. Pentachloro-<br>phenol (87-86-5)              | ×          |             |             | <10                           |                           |                        | н                     | ng/L             |         |                               |              |                       |
| 10A, Phenol<br>(108-95-2)                         | ×          |             |             | <10                           |                           |                        | Н                     | ng/L             |         |                               |              |                       |
| 11A, 2,4,6-Trichloro-<br>phenol (88-05-2)         | ×          |             |             | <10                           |                           |                        | П                     | µg/L             |         |                               |              |                       |
| L   | 100 07 00  |             |             |                               |                           |                        |                       |                  |         |                               |              |                       |

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|  | 2             | 2. MARK "X"     |         |                        | 3 FFFI LIENT              |                        |          | ATIMITS                               |         | 6 INTAKE (continue)           | V-       |
|--|---------------|-----------------|---------|------------------------|---------------------------|------------------------|----------|---------------------------------------|---------|-------------------------------|----------|
| 1. POLLUTANT   |               |                 |         |                        | b. MAXIMUM 30 DAY VALUE   | C. LONG TERM AVRG      |          | ÷.                                    |         | a LONG TERM                   | 11       |
|  | a.<br>TESTING | b,<br>REI IEVEN | b, c.   | a. MAXIMUM DAILY VALUE | $\rightarrow$             | VALUE (if available)   | ON TO    | N N N N N N N N N N N N N N N N N N N |         | ш                             | L C      |
| (if available)                                       | REQUIRED      | PRESENT         | ABSENT  | CONCENTRATION (2) MASS | SS CONCENTRATION (2) MASS | CONCENTRATION (2) MASS | ANALYSES | TRATION                               | b. MASS | (1)<br>CONCENTRATION (2) MASS | ANALYSES |
| GC/MS FRACTION - BASE/NEUTRAL COMPOUNDS              | - BASE/NE     | EUTRAL CC       | DMPOUND | S                      |                           |                        |          |                                       |         |                               |          |
| 1B. Acenaphthene<br>(83-32-9)                        | ×             |                 |         | <10                    |                           |                        | н        | ng/L                                  |         |                               |          |
| 2B. Acenaphtylene (208-96-8)                         |               |                 | ×       |                        |                           |                        |          |                                       |         |                               |          |
| 3B. Anthracene<br>(120-12-7)                         | ×             |                 |         | <10                    |                           |                        | н        | hg/L                                  |         |                               |          |
| 4B. Benzidine<br>(92-87-5)                           | ×             |                 |         | <10                    |                           |                        | П        | hg/L                                  |         |                               |          |
| 5B. Benzo (a)<br>Anthracene<br>(56-55-3)             | ×             |                 |         | <10                    |                           |                        | н        | hg/L                                  |         |                               |          |
| 6B. Benzo (a)<br>Pyrene (50-32-8)                    | ×             |                 |         | <10                    |                           |                        | н        | hg/L                                  |         |                               |          |
| 7B. 3,4-Benzo-<br>fluoranthene<br>(205-99-2)         | ×             |                 |         | <1.0                   |                           |                        | Н        | ng/L                                  |         |                               |          |
| 8B. Benzo (ghi)<br>Perylene (191-24-2)               |               |                 | ×       |                        |                           |                        |          |                                       |         |                               |          |
| 9B. Benzo (k)<br>Fluoranthene<br>(207-08-9)          | ×             |                 |         | <10                    |                           |                        | н        | hg/L                                  |         |                               |          |
| 10B. Bis (2-Chloro-<br>erhoxy) Methane<br>(111-91-1) |               |                 | ×       |                        |                           |                        |          |                                       |         |                               |          |
| 11B. Bis (2-Chloro-<br>ethyl) Ether<br>(111-44-4)    | ×             |                 |         | <10                    |                           |                        | н        | hg/L                                  |         |                               |          |
| 12B, Bis (2-<br>Chloroisopropyl)<br>Ether (102-80-1) | ×             |                 |         | <10                    |                           |                        | п        | hg/L                                  |         |                               |          |
| 13B. Bis (2-Ethyl-<br>hexyl) Phthalate<br>(117-81-7) |               |                 | ×       |                        |                           |                        |          |                                       |         |                               |          |
| 14B. 4-Bromophenyl<br>Phenyl Ether<br>(101-55-3)     |               |                 | ×       |                        |                           |                        |          |                                       |         |                               |          |
| 15B. Butyl Benzyl<br>Phthalate (85-68-7)             |               |                 | ×       |                        |                           |                        |          |                                       |         |                               |          |
| 16B. 2-Chloro-<br>naphthalene<br>(91-58-7)           | ×             |                 |         | <10                    |                           |                        | н        | T/bd                                  |         |                               |          |
| 17B. 4-Chloro-<br>phenyl Phenyl Ether<br>(7005-72-3) |               |                 | ×       |                        |                           |                        |          |                                       |         |                               |          |
| 18B. Chrysene<br>(218-01-9)                          | ×             |                 |         | <10                    |                           |                        | Н        | ng/L                                  |         |                               |          |
| 19B. Dibenzo (a.h)<br>Anthracene<br>(53-70-3)        | ×             |                 |         | <10                    |                           |                        | н        | hg/L                                  |         |                               |          |
| 20B. 1,2-Dichloro-<br>benzene (95-50-1)              | ×             |                 |         | <10                    |                           |                        | Н        | ng/L                                  |         |                               |          |
| 21B. 1,3-Di-chloro-<br>benzene (541-73-1)            | ×             |                 |         | <10                    |                           |                        | П        | ng/L                                  |         |                               |          |
| EPA Form 3510-2C (8-90)                              | (8-90)        |                 |         |                        | PAGE V-6                  | 9-^                    |          |                                       |         | CONTINUE ON PAGE V-7          | PAGE V-7 |

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| 2   |            | 2. MARK "X"         |                    |                     | 3. EFFLUENT             |                            |                       | 2 TINITS              | STIL    | S INTAKE (                 | in a                  |
|---|------------|---------------------|--------------------|---------------------|-------------------------|----------------------------|-----------------------|-----------------------|---------|----------------------------|-----------------------|
| 1. POLLUTANT<br>AND                                   | e          |                     |                    | MAXIMIM DAILY VALUE | b. MAXIMUM 30 DAY VALUE | c. LONG TERM AVRG.         | 20                    | 5                     |         | a. LONG TERM               | (puna)                |
| CAS NUMBER (if available)                             | TESTING    | BELIEVED<br>PRESENT | BELIEVED<br>ABSENT | ATION (2) M         | (1) (2) MASS            | (1) CONCENTRATION (2) MASS | d. NO. OF<br>ANALYSES | a. CONCEN-<br>TRATION | b. MASS | (1) CONCENTRATION (2) MASS | b. NO. OF<br>ANALYSES |
| GC/MS FRACTION - BASE/NEUTRAL COMPOUNDS (continued)   | V - BASE/N | JEUTRAL CO          | JMPOUNC            | OS (continued)      |                         |                            |                       |                       |         | 1                          | 1                     |
| 22B. 1,4-Dichloro-<br>benzene (106-46-7)              | ×          |                     |                    | <10                 |                         |                            | П                     | hg/L                  |         |                            |                       |
| 23B. 3,3-Dichloro-<br>benzidine (91-94-1)             | ×          |                     |                    | <20                 |                         |                            | п                     | hg/L                  |         |                            |                       |
| 24B. Diethyl<br>Phthalate (84-66-2)                   | ×          |                     |                    | <10                 |                         |                            | Т                     | ng/L                  |         |                            |                       |
| 25B. Dimethyl<br>Phthalate<br>(131 -11-3)             | ×          |                     |                    | <10                 |                         |                            | н                     | T/BH                  |         |                            |                       |
| 26B. Di-N-Butyl<br>Phthalate (84-74-2)                | ×          |                     |                    | <10                 |                         |                            | Н                     | ng/L                  |         |                            |                       |
| 27B. 2,4-Dinitro-<br>toluene (121-14-2)               | ×          |                     |                    | <10                 |                         |                            | ы                     | ng/L                  |         |                            |                       |
| 28B. 2,6-Dinitro-<br>toluene (606-20-2)               |            |                     | ×                  |                     |                         |                            |                       |                       |         |                            |                       |
| 29B. Di-N-Octyl<br>Phthalate (117-84-0)               |            |                     | ×                  |                     |                         |                            |                       |                       |         |                            |                       |
| 30B. 1,2-Diphenylhydrazine (as Azobenzene) (122-66-7) | ×          |                     |                    | <10                 |                         |                            | п                     | hg/L                  |         |                            |                       |
| 31B. Fluoranthene<br>(206-44-0)                       | ×          |                     |                    | <10                 |                         |                            | П                     | ng/L                  |         |                            |                       |
| 32B. Fluorene<br>(86-73-7)                            | ×          |                     |                    | <10                 |                         |                            | П                     | ng/L                  |         |                            |                       |
| 33B. Hexachloro-<br>benzene (118-74-1)                | ×          |                     |                    | < 1.0               |                         |                            | П                     | ng/L                  |         |                            |                       |
| 34B. Hexachloro-<br>butadiene (87-68-3)               | ×          |                     |                    | <1.0                |                         |                            | П                     | hg/L                  |         |                            |                       |
| 35B. Hexachloro-<br>cyclopentadiene<br>(77-47-4)      | ×          |                     |                    | <10                 |                         |                            | Н                     | пд/Гг                 |         |                            |                       |
| 36B Hexachloro-<br>ethane (67-72-1)                   | ×          |                     |                    | <10                 |                         |                            | П                     | ng/L                  |         |                            |                       |
| 37B. Indeno<br>(1,2,3-cd) Pyrene<br>(193-39-5)        | ×          |                     |                    | <20                 |                         |                            | Н                     | ng/L                  |         |                            |                       |
| 38B. Isophorone<br>(78-59-1)                          | ×          |                     |                    | <10                 |                         |                            | н                     | hg/L                  |         |                            |                       |
| 39B. Naphthalene<br>(91-20-3)                         | ×          |                     |                    | <1.0                |                         |                            | П                     | hg/L                  |         |                            |                       |
| 40B. Nitrobenzene<br>(98-95-3)                        | ×          |                     |                    | <10                 |                         |                            | П                     | T/6n                  |         |                            |                       |
| 41B. N-Nitro-<br>sodimethylamine<br>(62-75-9)         | ×          |                     |                    | <10                 |                         |                            | Н                     | ng/L                  |         |                            |                       |
| 42B. N-Nitrosodi-<br>N-Propylamine<br>(621-64-7)      | ×          |                     |                    | <10                 |                         |                            | П                     | ng/L                  |         |                            |                       |
| EPA Form 3510-2C (8-90)                               | (06-8)     |                     |                    |                     | PAGE V-7                | 7-7                        |                       |                       |         | CONTINUE                   | CONTINUE ON REVERSE   |

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|   |            | 2 MARK "X" |         |                        |           | 2 CEC LICKIT                           |   |                       |                  | -       |                               |          |
|---|------------|------------|---------|------------------------|-----------|--|---|-----------------------|------------------|---------|-------------------------------|----------|
| 1. POLLUTANT  |            |            |         |                        |           | S. E. LOEN                             |   |                       | 4. UNIIS         | 0       | 5. INTAKE (optional)          | 11)      |
|   | a.         | b. b.      | ي ز     | a. MAXIMUM DAILY VALUE | ILY VALUE | b. MAXIMUM 30 DAY VALUE (if available) | c. LONG TERM AVRG. VALUE (if available) |                       |                  |         | a. LONG TERM<br>AVERAGE VALUE |          |
| (if available)                                      | REQUIRED   | PRESENT    | ABSENT  | (1)<br>CONCENTRATION   | (2) MASS  | (1)<br>CONCENTRATION (2) MASS          | (1)<br>CONCENTRATION (2) MASS           | d. NO. OF<br>ANALYSES | a. CONCENTRATION | b. MASS | (1)<br>CONCENTRATION (2) MASS | ANALYSES |
| GC/MS FRACTION - BASE/NEUTRAL COMPOUNDS (continued) | - BASE/NE  | EUTRAL CC  | MPOUNDS | 3 (continued)          |           |  |   |                       |                  |         | 4                             |          |
| 43B. N-Nitro-<br>sodiphenylamine<br>(86-30-6)       | ×          |            |         | <10                    |           |  |   | п                     | µg/L             |         |                               |          |
| 44B. Phenanthrene (85-01-8)                         |            |            | ×       |                        |           |  |   |                       |                  |         |                               |          |
| 45B. Pyrene<br>(129-00-0)                           | ×          |            |         | <10                    |           |  |   | Н                     | ng/L             |         |                               |          |
| 46B. 1,2,4-Tri-<br>chlorobenzene<br>(120-82-1)      | ×          |            |         | <10                    |           |  |   | н                     | ng/L             |         |                               |          |
| GC/MS FRACTION - PESTICIDES                         | - PESTICII | DES        |         |                        |           |  |   |                       |                  |         |                               |          |
| 1P. Aldrin<br>(309-00-2)                            | ×          |            |         | <0.05                  |           |  |   | н                     | hg/L             |         |                               |          |
| 2P. α-BHC<br>(319-84-6)                             | ×          |            |         | <0.50                  |           |  |   | н                     | ng/L             |         |                               |          |
| 3P. β-BHC<br>(319-85-7)                             | ×          |            |         | <0.50                  |           |  |   | Н                     | ng/L             |         |                               |          |
| 4P. y-BHC (58-89-9)                                 | ×          |            |         | <0.50                  |           |  |   | 7                     | ng/L             |         |                               |          |
| 5P. 3-BHC<br>(319-86-8)                             | ×          |            |         | <0.50                  |           |  |   | н                     | ng/L             |         |                               |          |
| 6P. Chlordane<br>(57-74-9)                          | ×          |            |         | <0.20                  |           |  |   | н                     | ng/L             |         |                               |          |
| 7P. 4,4'-DDT<br>(50-29-3)                           | ×          |            |         | <0.10                  |           |  |   | н                     | ng/L             |         |                               |          |
| 8P. 4,4'-DDE<br>(72-55-9)                           | ×          |            |         | <0.10                  |           |  |   | Н                     | hg/L             |         |                               |          |
| 9P. 4,4DDD<br>(72-54-8)                             | ×          |            |         | <0.10                  |           |  |   | Н                     | hg/L             |         |                               |          |
| 10P. Dieldrin<br>(60-57-1)                          | ×          |            |         | <0.10                  |           |  |   | Н                     | hg/L             |         |                               |          |
| 11P. α-Enosulfan<br>(115-29-7)                      | ×          |            |         | <0.10                  |           |  |   | н                     | hg/L             |         |                               |          |
| 12P. β-Endosulfan<br>(115-29-7)                     | ×          |            |         | <0.10                  |           |  |   | н                     | ng/L             |         |                               |          |
| 13P. Endosulfan<br>Sulfate<br>(1031-07-8)           | ×          |            |         | <0.10                  |           |  |   | н                     | ng/L             |         |                               |          |
| 14P. Endrin<br>(72-20-8)                            | ×          |            |         | <0.10                  |           |  |   | П                     | hg/L             |         |                               |          |
| 15P. Endrin<br>Aldehyde<br>(7421-93-4)              | ×          |            |         | <0.10                  |           |  |   | н                     | µg/L             |         |                               |          |
| 16P. Heptachlor<br>(76-44-8)                        | ×          |            |         | <0.50                  |           |  |   | Н                     | ng/L             |         |                               |          |
| 00000   |            |            |         |                        |           |  |   |                       |                  |         |                               |          |

| CONTINUED TROM PAGE V-8                   | 0 1 1 0 1 0 |             |          |                        | V.       | VA0090981                              |             | 100                | -                                       |                       |                       |         |                               |                      |           |
|---|-------------|-------------|----------|------------------------|----------|--|-------------|--------------------|---|-----------------------|-----------------------|---------|-------------------------------|----------------------|-----------|
|   | LAGE V-2    |             |          |                        |          |  |             |                    |   |                       |                       |         |                               |                      |           |
|   | 2           | 2. MARK "X" |          |                        |          | 3. E                                   | 3. EFFLUENT |                    |   |                       | 4. UNITS              | ITS     | 5. INTA                       | 5. INTAKE (optional) | 0         |
| 1. POLLUTANT<br>AND                       |             | Ď.<br>Č     | ú        | a. MAXIMUM DAILY VALUE | LY VALUE | b. MAXIMUM 30 DAY VALUE (if available) | DAY VALUE   | c. LONG TERM AVRG. | AVRG.                                   | -                     |                       |         | a. LONG TERM<br>AVERAGE VALUE | ERM                  |           |
|   | REQUIRED    | BELIEVED    | BELIEVED | (1)<br>CONCENTRATION   | (2) MASS | (1)<br>CONCENTRATION                   | (2) MASS    | (1) d. NO. OF      | (2) MASS                                | d. NO. OF<br>ANALYSES | a. CONCEN-<br>TRATION | b. MASS | (1) (2) MACO                  | OS AMA CC.           | ANAL YSES |
| GC/MS FRACTION - PESTICIDES (continued)   | - PESTICIL  | DES (contim | (pai     |                        |          |  |             |                    | 200000000000000000000000000000000000000 |                       | 1                     |         |                               | CCAM (2)             |           |
| 17P. Heptachlor<br>Epoxide<br>(1024-57-3) | ×           |             |          | <0.50                  |          |  |             |                    |   | н                     | hg/L                  |         |                               |                      |           |
| 18P. PCB-1242<br>(53469-21-9)             | ×           |             |          | <1.00                  |          |  |             |                    |   | 2                     | µg/L                  |         |                               |                      |           |
| 19P. PCB-1254<br>(11097-69-1)             | ×           |             |          | <1.00                  |          |  |             |                    |   | 2                     | hg/L                  |         |                               |                      |           |
| 20P. PCB-1221<br>(11104-28-2)             | ×           |             |          | <1.00                  |          |  |             |                    |   | 23                    | hg/L                  |         |                               |                      |           |
| 21P. PCB-1232<br>(11141-16-5)             | ×           |             |          | <1.00                  |          |  |             |                    |   | 23                    | µg/L                  |         |                               |                      |           |
| 22P. PCB-1248<br>(12672-29-6)             | ×           |             |          | <1.00                  |          |  |             |                    |   | 2                     | µg/L                  |         |                               |                      |           |
| 23P. PCB-1260<br>(11096-82-5)             | ×           |             |          | <1.00                  |          |  |             |                    |   | 2                     | µg/L                  |         |                               |                      |           |
| 24P. PCB-1016<br>(12674-11-2)             | ×           |             |          | <1.00                  |          |  |             |                    |   | 2                     | ng/L                  |         |                               |                      |           |
| 25P. Toxaphene<br>(8001-35-2)             | ×           |             |          | <5.00                  |          |  |             |                    |   | П                     | hg/L                  |         |                               |                      |           |

EPA I.D. NUMBER (copy from Item 1 of Form 1) OUTFALL NUMBER

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VA0090981

Form Approved. OMB No. 2040-0086 Approval expires 5-31-92

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U.S. Environmental Protection Agency Washington, DC 20460

#### Application for Permit to Discharge Storm Water Discharges Associated with Industrial Activity

#### Paperwork Reduction Act Notice

Public reporting burden for this application is estimated to average 28.6 hours per application, including time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding the burden estimate, any other aspect of this collection of information, or suggestions for improving this form, including suggestions which may increase or reduce this burden to: Chief, Information Policy Branch, PM-223, U.S. Environmental Protection Agency, 1200 Pennsylvania Avenue, NW, Washington, DC 20460, or Director, Office of Information and Regulatory Affairs, Office of Management and Budget, Washington, DC 20503.

| I. Outfall Location       |                 |                  |                |               |              |            | Constitution of the state of th | į |
|---------------------------|-----------------|------------------|----------------|---------------|--------------|------------|--|---|
| For each outfall, list th | ne latitude and | d longitude of i | ts location to | the nearest 1 | 5 seconds an | d the name | of the receiving water.  |   |
| A. Outfall Number (list)  |                 | B. Latitude      |                |               | . Longitude  |            | D. Receiving Water (name)  |   |
| 002                       | 36.00           | 58.00            | 10.41          | 77.00         | 24.00        | 11.75      | GALLEY SWAMP   | - |
|                           |                 |                  |                |               |              |            | see Appendix 1, Fig. 1 for location map  | - |
|                           |                 |                  |                |               |              |            |  | _ |
|                           |                 |                  |                |               |              |            |  |   |
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| II. Improvements          |                 |                  |                |               |              |            |  |   |

A. Are you now required by any Federal, State, or local authority to meet any implementation schedule for the construction, upgrading or operation of wastewater treatment equipment or practices or any other environmental programs which may affect the discharges described in this application? This includes, but is not limited to, permit conditions, administrative or enforcement orders, enforcement compliance schedule letters, stipulations, court orders, and grant or loan conditions.

| Identification of Conditions,<br>Agreements, Etc. |                            | 2. Affected Outfalls |                              | 4. F<br>Complia | Final     Compliance Date |  |
|---|----------------------------|----------------------|------------------------------|-----------------|---------------------------|--|
| Agreements, Etc.                                  | number source of discharge |                      | Brief Description of Project | a. req.         | b. proj.                  |  |
| NA  | NA                         | NA                   | NA                           |                 |                           |  |
|   |                            |                      |                              |                 |                           |  |
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|   |                            |                      |                              |                 |                           |  |

B: You may attach additional sheets describing any additional water pollution (or other environmental projects which may affect your discharges) you now have under way or which you plan. Indicate whether each program is now under way or planned, and indicate your actual or planned schedules for construction

#### III. Site Drainage Map

Attach a site map showing topography (or indicating the outline of drainage areas served by the outfalls(s) covered in the application if a topographic map is unavailable) depicting the facility including: each of its intake and discharge structures; the drainage area of each storm water outfall; paved areas and buildings within the drainage area of each storm water outfall, each known past or present areas used for outdoor storage of disposal of significant materials, each existing structural control measure to reduce pollutants in storm water runoff, materials loading and access areas, areas where pesticides, herbicides, soil conditioners and fertilizers are applied; each of its hazardous waste treatment, storage or disposal units (including each area not required to have a RCRA permit which is used for accumulating hazardous waste under 40 CFR 262.34); each well where fluids from the facility are injected underground; springs, and other surface water bodies which received storm water discharges from the facility. Please see Attachment ()

|                         | om the Front  |   |                                       |   |                  |                                    |
|-------------------------|---|---|---------------------------------------|---|------------------|------------------------------------|
| IV. Narra               | tive Description of Pollutant Sc  | urces   |                                       |   |                  |                                    |
| A. For eac<br>drained   | h outfall, provide an estimate of the area (include<br>by the outfall.  | units) of imperious surface                   | es (including paved                   | areas and building roofs) drained to the outfal | I, and an estima | ate of the total surface area      |
| Outfall<br>Number       | Area of Impervious Surface (provide units)  | Total Area Drained (provide units)            | Outfall<br>Number                     | Area of Impervious Surface (provide units)      |                  | Total Area Drained (provide units) |
| 002                     | 5.5 ACRES   | 11.85 ACRES                                   |                                       |   |                  |                                    |
| storm v<br>applied      |   | disposal: past and pre-                       | sent materials ma                     | anagement practices employed to minir           | mize contact     | by those meterials with            |
| C. For ea               | FER TO APPENDIX 2, DOCUMENT 2.  | cription of existing stru                     | ictural and nonstr                    | uctural control measures to reduce po           | llutants in sto  | rm water runoff; and a             |
| of any                  | otion of the treatment the storm water rece<br>solid or fluid wastes other than by discharg                                     | ge.   | edule and type of                     | maintenance for control and treatment           | measures ar      |                                    |
| Number                  |   | Tı  | reatment                              |   |                  | List Codes from<br>Table 2F-1      |
| 002                     | Outfall 002 is fed from the Ms<br>approximately 2.5 hours of ret<br>year storm event (see Appendix<br>level spreader with weir. | ention and settli                             | ng time for                           | suspended solids during the 1                   | hr/25            | 1-0                                |
| V. Nonsto               | rmwater Discharges  | The street                                    |                                       |   |                  |                                    |
| A. I certify nonsto     | runder penalty of law hat the outfall(s) co<br>rmwater discharged from these outfall(s) a                                       | vered by this applicatio                      | n have been test                      | ed or evaluated for the presence of nor         | nstormwater o    | discharges, and that all           |
| Name and C              | Official Title (type or print)  NR SALO  PLES INDUST  |   |                                       | om 20 of from 22 approaches for the c           | Date Sig         | 121/07.                            |
| B. Provide              | e a description of the method used, the dat   | e of any testing, and the                     | e onsite drainage                     | points that were directly observed durin        | ng a test.       |                                    |
|                         | s water lines are accounted for   |   |                                       |   | <u>3</u>         |                                    |
| VI. Signifi             | cant Leaks or Spills  |   | - jakener (s. line                    |   |                  |                                    |
| Provide e:<br>approxima | xisting information regarding the history at<br>the date and location of the spill or leak, an                                  | of significant leaks or d the type and amount | spills of toxic or of material releas | hazardous pollutants at the facility in ed.     | the last thre    | e years, including the             |
| NA                      |   |   |                                       |   |                  |                                    |
|                         |   |   |                                       |   |                  |                                    |

#### Continued from Page 2

EPA ID Number (copy from Item 1 of Form 1) VA0090981

| VII. Discharge Information  |  |   |  |  |  |  |  |  |  |
|---|--|---|--|--|--|--|--|--|--|
| A, B, C, & D: See instructions before proceeding. Complete one set of tables for each outfall. Annotate the outfall number in the space provided.  Table VII-A, VII-B, VII-C are included on separate sheets numbers VII-1 and VII-2.   |  |   |  |  |  |  |  |  |  |
| Potential discharges not covered by<br>currently use or manufacture as an in  | analysis – is any toxic pollutant listed in table 2 termediate or final product or byproduct?  | F-2, 2F-3, or 2F-4, a substance or a                        | component of a substance which you                       |  |  |  |  |  |  |
| Yes (list all such pollutants   |  | No (go to Section IX)                                       |  |  |  |  |  |  |  |
|   | itanium, Sulfite*, Sulfate, Zirconium**, Oil and Grease.   |   |  |  |  |  |  |  |  |
| * Sulfite could potentially form from the use of Sulfuric Acid in the ZFP. No testing has been conducted for sulfite as it is believed absent. Sulfite readily oxidizes to sulfate in the oxygenated environment typical of surface waters. Testing has been conducted for the presence of sulfate. |  |   |  |  |  |  |  |  |  |
| ** Zirconium is a constituent<br>testing has been conduted for Zi<br>at the MSP and ZFP.  | of the zircon sand product produced by rconium. It is believed absent due to   | by the MSP as well as the raw the insolubility of zircon to | mineral sand concentrate. No under the condtions present |  |  |  |  |  |  |
| VIII. Biological Toxicity Testing   |  |   |  |  |  |  |  |  |  |
| Do you have any knowledge or reason to<br>relation to your discharge within the last 3  | believe that any biological test for acute or chroni years?  | ic toxicity has been made on any of you                     | ur discharges or on a receiving water in                 |  |  |  |  |  |  |
| Yes (list all such pollutants   | The state of the s | ✓ No (go to Section IX)                                     |  |  |  |  |  |  |  |
| Yes (list the name, address,  | N VII performed by a contract laboratory or consulting and telephone number of, and pollutants laboratory or firm below)   | ng firm?  |  |  |  |  |  |  |  |
| A. Name   | B. Address   | C. Area Code & Phone No.                                    | D. Pollutants Analyzed                                   |  |  |  |  |  |  |
| Primary Laboratories  FROEHLING & ROBERTSON, INC  | 7423 Lee Davis Road<br>Mechanicsville, VA 23111<br>3015 DUMBARTON ROAD   | 804-559-9004<br>804-264-2701                                | See APPENDIX 3, ITEM 3                                   |  |  |  |  |  |  |
| directly responsible for gathering the info<br>there are significant penalties for submittin  A. Name & Official Title (Type Or Print)  | ument and all attachments were prepared under id evaluate the information submitted. Based on in mation, the information submitted is, to the best ig false information, including the possibility of fine   | ny inquiry of the person or persons who                     | manage the system or those persons                       |  |  |  |  |  |  |
| ALLAN R SALE  C. Signature  | - PZESIDENT  | 1 434 348 4   | 1302   |  |  |  |  |  |  |
| o. orginature 1111  |  | D. Date Signed 7 /21/0 7                                    |  |  |  |  |  |  |  |

#### VII. Discharge information (Continued from page 3 of Form 2F)

Part A - You must provide the results of at least one analysis for every pollutant in this table. Complete one table for each outfall. See instructions for additional details.

|  |  | num Values<br>ude units)   | Average Values (include units)                     |                            | Number                           |                                     |  |
|--|--|----------------------------|--|----------------------------|----------------------------------|-------------------------------------|--|
| Pollutant<br>and<br>CAS Number<br>(if available) | Grab Sample<br>Taken During<br>First 20<br>Minutes | Flow-Weighted<br>Composite | Grab Sample<br>Taken During<br>First 20<br>Minutes | Flow-Weighted<br>Composite | of<br>Storm<br>Events<br>Sampled | Sources of Pollutants               |  |
| Oil and Grease                                   | BQL mg/L   | N/A                        | na   | na                         | 2.00                             | na                                  |  |
| Biological Oxygen<br>Demand (BOD5)               | <2.0 mg/L  | na                         | na   | na                         | 2.00                             | Unknown                             |  |
| Chemical Oxygen<br>Demand (COD)                  | 37 mg/L  | na                         | 25.5 mg/L  | na                         | 2.00                             | Unknown                             |  |
| Total Suspended<br>Solids (TSS)                  | 42.4 mg/L  | na                         | 11.0 mg/L  | na                         | 16.00                            | Entrained solids in stormwater flow |  |
| Total Nitrogen                                   | 1.1 mg/L   | na                         | <1.1 mg/L  | na                         | 2.00                             | Unknown                             |  |
| Total Phosphorus                                 | 0.04 mg/L  | na                         | .035 mg/L  | na                         | 2.00                             | Unknown                             |  |
| pН   | Minimum 5.10                                       | Maximum 8.54               | Minimum  | Maximum                    |                                  | Control of the control              |  |

Part B – List each pollutant that is limited in an effluent guideline which the facility is subject to or any pollutant listed in the facility's NPDES permit for its process wastewater (if the facility is operating under an existing NPDES permit). Complete one table for each outfall. See the instructions for additional details and requirements.

|  | (inclu   | um Values<br>ide units)    | Avera<br>(inc.                                     | age Values<br>lude units)  | Number                           |                       |  |
|--|--|----------------------------|--|----------------------------|----------------------------------|-----------------------|--|
| Pollutant<br>and<br>CAS Number<br>(if available) | Grab Sample<br>Taken During<br>First 20<br>Minutes | Flow-Weighted<br>Composite | Grab Sample<br>Taken During<br>First 20<br>Minutes | Flow-Weighted<br>Composite | of<br>Storm<br>Events<br>Sampled | Sources of Pollutants |  |
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#### Continued from the Front

Part C - List each pollutant shown in Table 2F-2, 2F-3, and 2F-4 that you know or have reason to believe is present. See the instructions for additional details and requirements. Complete one table for each outfall.

|  | (incl  | Maximum Values<br>(include units) |  | erage Values<br>nclude units) | Number                           |                               |
|--|--|-----------------------------------|--|-------------------------------|----------------------------------|-------------------------------|
| Pollutant<br>and<br>CAS Number<br>(if available) | Grab Sample<br>Taken During<br>First 20<br>Minutes | Flow-Weighted<br>Composite        | Grab Sample<br>Taken During<br>First 20<br>Minutes | Flow-Weighted<br>Composite    | of<br>Storm<br>Events<br>Sampled | Sources of Pollutants         |
| Silver   | BQL mg/L   | na                                | na   | na                            | 1.00                             | na                            |
| Alkalinity                                       | 15 mg/L  | na                                | na   | na                            | 1.00                             | Hydrated lime                 |
| Ammonia-N  | 0.10 mg/L  | na                                | na   | na                            | 1.00                             | Unknown                       |
| Arsenic  | BQL mg/L   | na                                | na   | na                            | 1.00                             | na                            |
| Barium   | BQL mg/L   | na                                | na   | na                            | 1.00                             | na                            |
| Cadmium  | BQL mg/L   | na                                | na   | na                            | 1.00                             | na                            |
| Chromium   | BQL mg/L   | na                                | na   | na                            | 1.00                             | na                            |
| Iron   | 1.9 mg/L   | na                                | na   | na                            | 1.00                             | ilmenite, scrap metal, zircon |
| Mercury  | BQL mg/L   | na                                | na   | na                            | 1.00                             | na                            |
| Potassium  | 3.4 mg/L   | na                                | na   | na                            | 1.00                             | Unknown                       |
| Sodium   | 11 mg/L  | na                                | na   | na                            | 1.00                             | Unknown                       |
| Nitrite  | BQL mg/L   | na                                | na   | na                            | 1.00                             | na                            |
| Nitrate  | 0.14 mg/L  | na                                | na   | na                            | 1.00                             | Unknown                       |
| Lead   | BQL mg/L   | na                                | na   | na                            | 1.00                             | na                            |
| Selenium   | BQL mg/L   | na                                | na   | na                            | 1.00                             | na                            |
| Sulfate  | 2.8 mg/L   | na                                | na   | na                            | 1.00                             | Unknown                       |
| roc  | 9.7 mg/L   | na                                | na   | na                            | 1.00                             | Unknown                       |
| Titanium   | BQL mg/L   | na                                | na   | na                            | 1.00                             | na                            |
|  |  |                                   |  |                               |                                  |                               |
|  |  |                                   |  |                               |                                  |                               |
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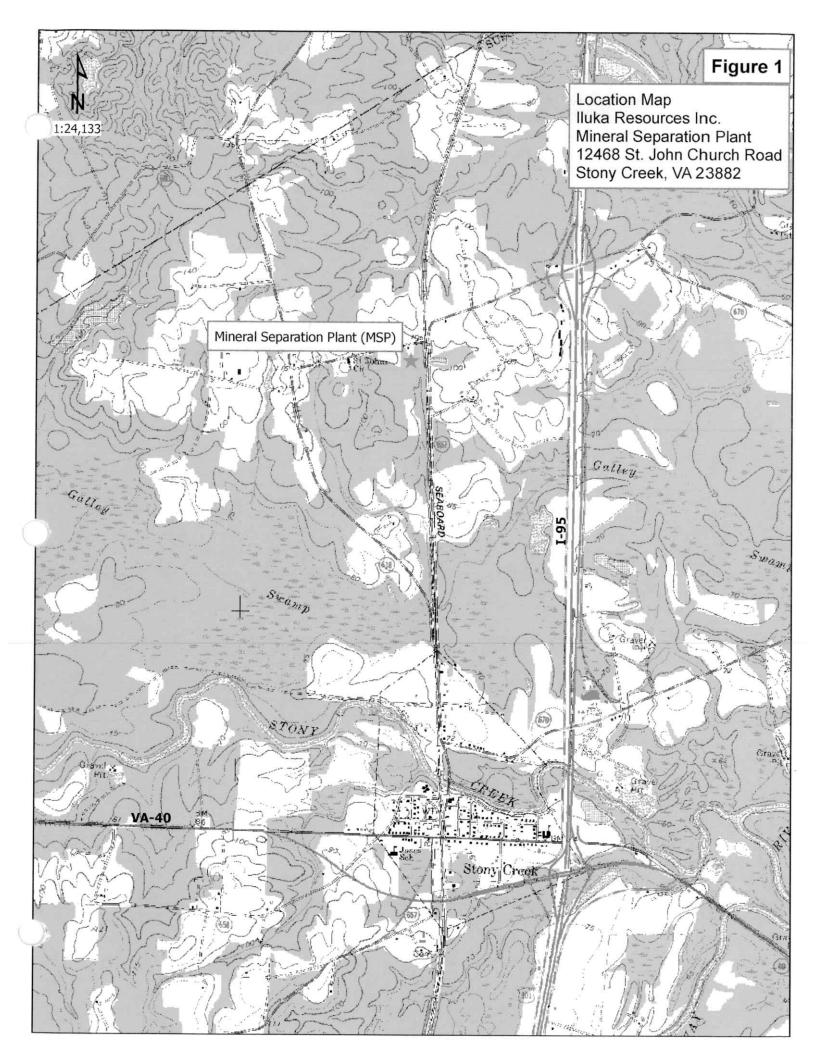
Part D - Provide data for the storm event(s) which resulted in the maximum values for the flow weighted composite sample.

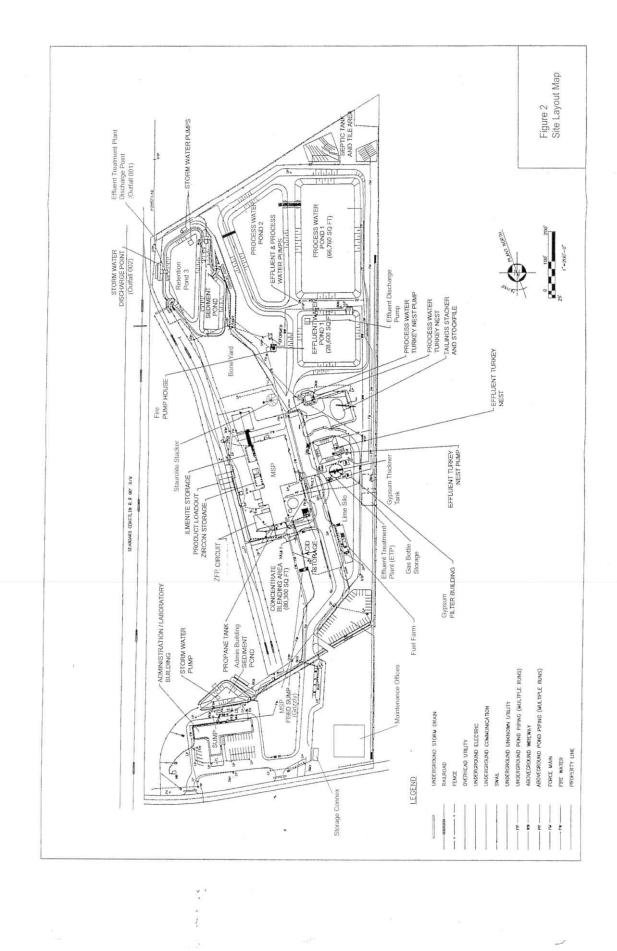
| 1.<br>Date of<br>Storm<br>Event | 2.<br>Duration<br>of Storm Event<br>(in minutes) | 3.<br>Total rainfall<br>during storm event<br>(in inches) | 4. Number of hours between beginning of storm measured and end of previous measurable rain event | 5.  Maximum flow rate during rain event (gallons/minute or specify units) | 6.<br>Total flow from<br>rain event<br>(gallons or specify units) |
|---------------------------------|--|---|--|---|---|
| a .                             | na   | na  | na   | na  | na  |

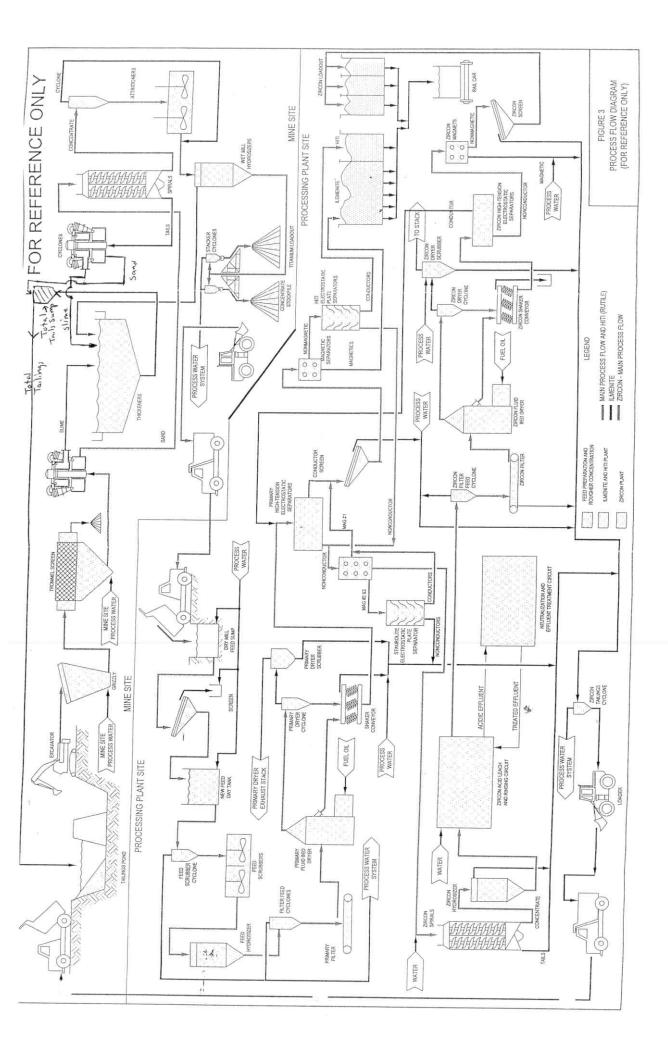
<sup>7.</sup> Provide a description of the method of flow measurement or estimate.

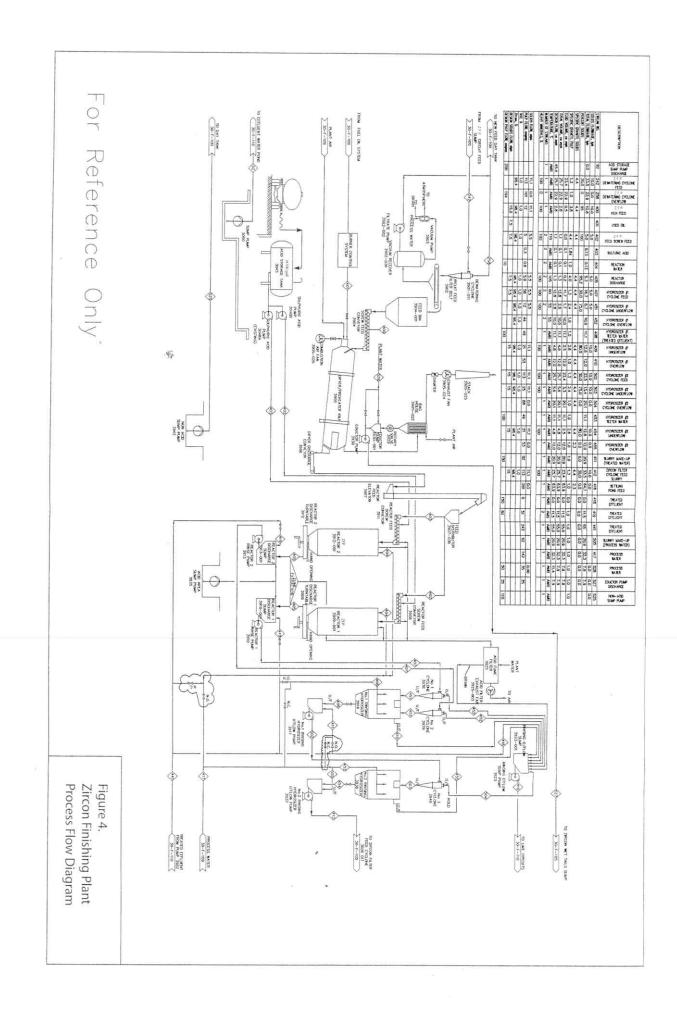
A hand held electronic flow meter is used to obtain the velocity of the flow leaving the outfall, this is multiplied by the depth of the water passing through the outfall to obtain an estimated volume of discharge.

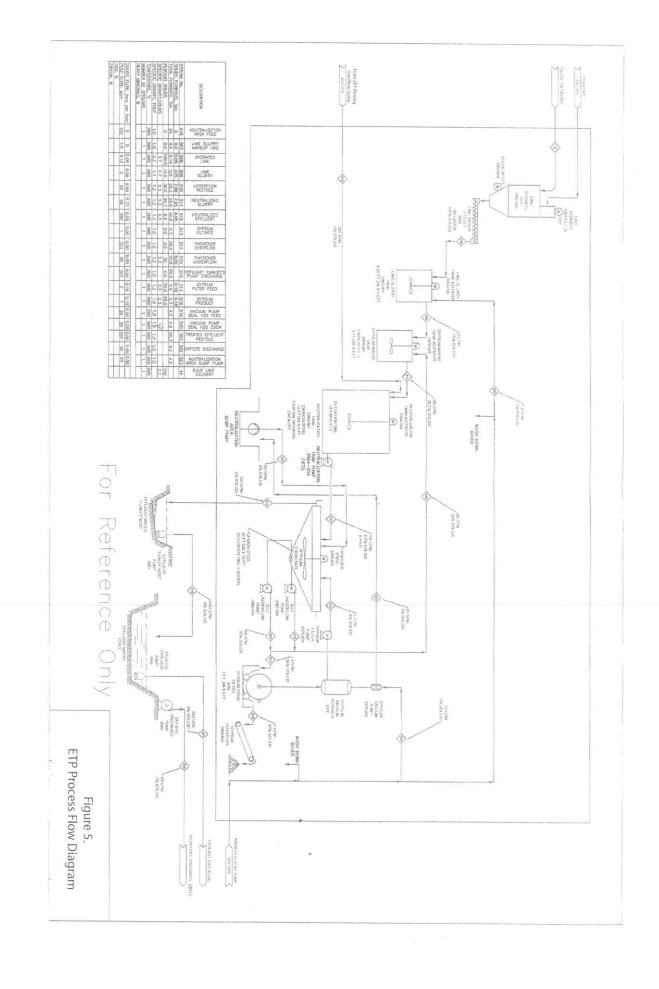
# APPENDIX 1 MAPS AND FIGURES











# APPENDIX 2 DOCUMENTS

DOCUMENT 1

## DOCUMENT 1: PROCESS OVERVIEW AND WASTEWATER TREATMENT DESCRIPTION

#### 1.0 OVERVIEW OF OPERATIONS AND WASTEWATER SOURCES

Iluka Resources' 36.6 acre Processing site located in Sussex County, near Stony Creek (see Appendix 1, Figure 1) includes the Mineral Separation Plant (MSP), Administrative/Laboratory Building, and storm water management system (see Appendix 1, Figure 2). Major operations involved in the processing of mineral sands are conducted in and around the MSP and include the following:

- Mineral Sand Concentrate Storage and Plant Feed
- Drying of Concentrate Feed
- · Ilmenite and HiTi Circuit
- Zircon Recovery Circuit
- Zircon Finishing Plant Circuit
- Staurolite Removal Circuit
- Zircon Finishing Plant Effluent Treatment Plant (ETP)
- Tailings Removal

A process flow diagram that shows the major steps in the production of titanium-bearing and zircon mineral sand concentrate from the mine site to the production of finished products at the MSP is included in Appendix 1, Figure 3. The MSP typically operates continuously, 24 hours a day, seven days a week.

#### 1.1 Mineral Sand Concentrate Storage and Plant Feed

At the mine and Mine Concentrator sites, mineral sands ore is excavated from relic beach ridge deposits by track-hoe excavators, slurried, and concentrated using various wet gravity separation processes. Approximately 10 percent by weight of the ore body is removed from the excavated material as mineral sand concentrate and the remaining 90 percent clay and sand tailings are returned to the tailings pit in the mine area, graded and reclaimed in accordance with all necessary permits. The mineral sand concentrate is pumped from the Mine Concentrator building to a concentrate stacker where excess water is removed and returned to the Mine Concentrator process water pond. The raw

mineral sand concentrate, which consists of approximately 90 percent mineral sands, is then hauled by truck approximately 14 miles to the MSP for final separation into the ilmenite and zircon mineral sands products. After weigh-in on the scales at the entrance to the MSP, the trucks dump the raw concentrate in an approximately 1.85-acre storage stockpile area located immediately north of the MSP. Raw concentrate is removed from the stockpile area and fed into the MSP's grizzly feed sump (see Appendix 1, Figure 2 for location) by the site's Volvo front end loader.

#### 1.2 Drying of Concentrate Feed

Drying is required to reduce moisture content to less than 0.1 percent by weight prior to electrostatic and electromagnetic processing for separation of titanium bearing-minerals (ilmenite, rutile and leucoxene) from zircon and non-conducting gangue minerals. The mineral sand concentrate loaded into the outside grizzly feed sump is slurried with clean water from the Process Water Pond, screened, and pumped into the 1,600-ton new feed day tank. Mineral slurry from the day tank is pumped to the scrubber rinse circuit and then to the filter feed cyclones to remove excess water prior to additional dewatering on a belt filter. The belt reduces the moisture content of the feed to approximately 5 percent by weight. The filtrate liquid is pumped to the process water system for reuse. The filter cake from the belt press is temporarily stored in a bin and then screw fed into a fluid-bed dryer to reduce moisture content to less than 0.1 percent by weight.

#### 1.3 Ilmenite and HiTi Circuit

After the concentrate is sufficiently dried, the material is screened to remove oversize material, which is sent to the tailings stockpile. The underflow from the screen is fed into high-tension electrostatic (HT) separators, which separate the electrically conductive ilmenite and rutile from the non-conductive zircon and gangue minerals. The conductive minerals are screened and the oversize material is slurried and pumped to the tailings stockpile. The material passing through the screen is sent through a series of magnets to separate the more magnetic titanium-bearing mineral ilmenite from the less magnetic titanium-bearing minerals (rutile and leucoxene) commonly referred to as HiTi. The

magnetic titanium-bearing minerals collected from each magnet is combined and conveyed to the ilmenite product storage bin prior to shipment. The less magnetic HiTi minerals pass through a series of electrostatic plate separators to remove any residual non-conductive material before being combined with the ilmenite product stream prior to storage in the ilmenite product storage bin. Various properties of the minerals processed at the MSP can be found in Table 1.

Table 1: Properties of Minerals

|                     | Ilmenite              | Rutile                        | Leucoxene                             | Zircon                        |
|---------------------|-----------------------|-------------------------------|---------------------------------------|-------------------------------|
| Chemical Formula    | FeTiO <sub>3</sub> ,  | TiO <sub>2</sub>              | TiO <sub>2</sub> + FeTiO <sub>3</sub> | ZrSiO <sub>4</sub>            |
| Specific Gravity    | 4.75                  | 4.22                          | 3.95                                  | 4.69                          |
| Magnetic Properties | Conductor<br>Magnetic | Conductor<br>Non-<br>Magnetic | Conductor<br>Weakly Magnetic          | Non-Conductor<br>Non-Magnetic |

#### 1.4 Zircon Recovery and Finishing Plant Circuits

Because of strict customer quality control requirements regarding contaminants in the final zircon product, Iluka uses only clean water from Process Water Pond 2 for the final rinsing step in Rinsing Hydrosizer No. 2. Treated effluent water from the ETP is used in all other rinsing steps in the Zircon Finishing Plant (ZFP). The overflow from Rinsing Hydrosizer No. 2 empties into the Rinsing Sump. The cleaned zircon concentrate underflow is discharged into a sump and then pumped into a final dewatering cyclone. The dewatered zircon concentrate is conveyed to a vacuum filter belt for additional dewatering and then to the zircon fluid bed dryer for removal of any remaining moisture. A process flow diagram for the ZFP is included in Appendix 1 as Figure 4.

The dried zircon concentrate is screened to remove oversized material and fed into a secondary three-stage HT separator to remove any residual conductive material. This conductive material is returned to the primary HT circuit to recover any titanium-bearing minerals. The non-conductive material is passed through a high-intensity magnetic separator circuit to remove any residual magnetic material. The magnetic material is sent to the tailings stockpile while the non-magnetic material (primarily zircon) is screened and conveyed to the zircon product storage bins for shipment.

#### 1.5 Staurolite Removal Circuit

The Staurolite Removal Circuit is contained within the MSP. The non-conductor stream from the Ilmentite and HiTi Circuit is combined with the non-conductor product from the initial electrostatic separation process to form the feed for the Staurolite Removal Circuit. An elevator and conveyor move the feed to a set of rare earth roll magnetic separators (RERMS), which produce separate mag1, mag2 and non-mag material streams. The top roll, with its lower magnetic rating, recovers highly magnetic material (mag1); primarily misplaced ilmenite. The mag1 material is directed to the base of the elevator that feeds the conductor screens. The second and third rolls recover paramagnetic material (mag2) that is passed through an electrostatic separator to scavenge any misplaced conductor material. The paramagnetic conductor stream is sent to the conductor screen elevator along with the mag1 product. The paramagnetic non-conducting staurolite-containing material (non-mag) is presently sent to the conductor oversize sump and from there to the staurolite stacker for disposal as tailings. However, the staurolite-containing material is available for packaging should a market for it be discovered. The non-magnetic stream from the RERMS can be directed to a surge bin for storage or as feed to the Zircon Recovery Circuit.

#### 1.6 Tailings

Tailings are slurried and pumped to the tailings stacker located immediately west of the MSP (see Appendix 1, Figure 2 for location). Excess water from the tailings stacker drains into the Tailings Turkey Nest. An electric-powered pump then transfers water from the Tailings Turkey Nest to the Process Water Pond for reuse. The stacked tailings are loaded into dump trucks and hauled back to the mine sites where they are backfilled into the mine pits and graded for reclamation.

#### 2.0 ZIRCON FINISHING PLANT WATER QUALITY

#### 2.1 Effluent Water Quality

The mineral sand concentrate processed at the MSP includes the titanium-bearing minerals ilmenite, rutile, and leucoxene, in addition to zircon and miscellaneous gangue minerals. By taking advantage of their differing physical properties (see Table 1), these minerals are separated from each other using non-chemical techniques including various wet gravity methods, high-tension electrostatic separation, and magnetic separation.

The titanium-bearing minerals are separated from the zircon prior to the zircon finishing circuit using high-tension electrostatic separators as described in Section 2.3.

Therefore, very little titanium-bearing material is expected to be present during this step.

#### 2.1.1 Analytical Testing

Lab reports resulting from analytical testing of the effluent stream are included in this renewal application in Appendix 3, as Items 1, 2 and 3.

### 3.0 ZIRCON EFFLUENT WATER TREATMENT PLANT

The Effluent Treatment Plant (ETP) system incorporates the following general processes to reduce the sulfate, iron, and trace metals expected in the influent stream.

- · Neutralization and precipitation
- · Clarification and sludge thickening
- ZFP treated process water storage, reuse, and discharge
- · Gypsum sludge dewatering and disposal

The ETP utilizes hydrated lime as the neutralizing and precipitating agent. The system is based on EIMCO Process Equipment Company's (EIMCO) precipitant stabilization solid recycle (PSSR) system. The process water treatment plant is located on concrete

pads to the west of the MSP (see Appendix 1, Figure 2). Drains in the concrete pads allow for spills and precipitation to gravity flow to the concrete Neutralization Area Sump located underneath the Gypsum Thickener where the recovered water is pumped back into the treatment system. A process flow diagram for the ETP can be found in Appendix 1, Figure 5.

#### 3.1 Lime Storage, Slurry Generation, and Neutralization

Lime storage, lime slurry mixing, and neutralization is performed in a portion of the ETP enclosed in a steel silo located west of the MSP on a concrete pad. Hydrated lime powder is hauled in bulk tanker trucks to the ETP where a pneumatic transfer system is used to convey the hydrated lime powder from the tanker truck to the Lime Storage Bin, situated in the upper portion of the silo. This storage bin is equipped with air pollution control devices to capture fugitive lime dust generated during transfer operations.

The Lime Slurry Tank, located underneath the Lime Storage Bin, is constructed of high carbon steel with a nominal capacity of 477 gallons and a working capacity of 371 gallons. The contents of the Lime Slurry Tank are continuously mixed with a stainless steel agitator. A Programmable Logic Control (PLC) operated variable frequency drive controls the lime screw feeder from the Lime Storage Bin, thus allowing for control of the lime slurry concentration. The lime slurry overflows into the Gypsum Mixing Tank, which is located adjacent to the Lime Silo.

The Gypsum Mixing Tank is made of high carbon steel with a nominal capacity of 420 gallons and a working capacity of 397 gallons. In this tank, lime slurry from the Lime Slurry Tank is seeded with approximately 97 gallons per minute of recycled gypsum underflow from the Gypsum Thickener while being continuously mixed with a stainless steel agitator. The contents of the Gypsum Mixing Tank then overflow into the Neutralization Tank.

The Neutralization Tank is located in the Lime Silo underneath the Lime Slurry Tank. It is constructed of high carbon steel with a nominal capacity of 6,770 gallons and a

working capacity of 5,500 gallons. The Rinsing Overflow Sump Pump in the ZFP transfers approximately 285 gallons per minute of acidic wastewater to the Neutralization Tank where it is neutralized by chemical reactions with the lime and gypsum slurry from the Gypsum Mixing Tank. The contents of the Neutralization Tank are continuously mixed with a stainless steel agitator, promoting the neutralization reactions. Retention time in the Neutralization Tank is approximately 15 minutes. The neutralized effluent water and suspended solids are then pumped from the Neutralization Tank to the Gypsum Thickener using a variable frequency drive, centrifugal pump rated for 400 gallons per minute. An ultrasonic level indicator mounted on the Neutralization Tank controls the pump speed and triggers a low-level alarm when the liquid level in the tank falls to 2.5 feet above the tank bottom and a high-level alarm at 7.0 feet above the tank bottom. The PLC receives the alarm information and sends a signal to the ETP's Motor Control Center (MCC) and the pump speed is adjusted accordingly. A pH indicator/controller continuously monitors the pH of the treated effluent leaving the Neutralization Tank and relays the data to the ETP's PLC where the pH data is compared to the pH setpoint. The setpoint is adjusted to maintain the effluent pH between 6 and 9 standard units. When the pH of the effluent falls below the setpoint the PLC signals the ETP's MCC to activate the Lime Storage Bin vibrator, the lime screw feeder, and plant water supply valve to mix additional lime slurry to be fed into the Neutralization Tank.

#### 3.2 Clarification and Sludge Thickening

The Gypsum Thickener is made of high carbon steel with a nominal capacity of 142,750 gallons, a working capacity of 136,800 gallons, and detention time of approximately 5.7 hours. It is set above grade and is supported by steel piers on a concrete pad. Neutralized effluent water enters the tank through the Thickener's center well, where the water and suspended solids are discharged in a downward direction below the liquid level in the tank. Velocity of the flow decreases after leaving the center well, allowing suspended solids to settle. Clarified, treated water flows upward and outward from the center well to peripheral weirs along the outside tank wall where it then flows into a collection launder. Heavier suspended solids entering the thickener are the first to settle

and accumulate on the floor of the thickener. Less dense suspended solids may be carried upward for a short distance with the water until the velocity of the flow falls below the settling velocity of the particles. The sludge blanket that forms at this equilibrium point acts as a filter, trapping smaller, less dense particles. Eventually, these particles agglomerate and slowly fall, to be removed with the denser gypsum sludge. A set of motorized rakes slowly rotates on the floor of the Thickener, moving the settled sludge downward along the sloped tank bottom and into the sludge well. The 30 percent solids in the sludge well are then pumped by one of two Thickener Underflow Pumps rated for 100 gallons per minute. One pump is installed as a spare while the other pump continuously recycles approximately 97 gallons per minute of 30 percent solids to the Gypsum Mixing Tank. Approximately 3 gallons per minute of 30 percent solids are fed into the Vacuum Drum Filter for gypsum sludge dewatering and removal.

#### 3.3 Treated Water Storage and Disposal

The treated effluent water from the Gypsum Thickener is piped from the collection launder to the Effluent Turkey Nest for temporary storage. This 10-foot deep pond is constructed to hold approximately 230,000 gallons of treated effluent water with a minimum freeboard of 2 feet. The Turkey Nest is lined with an 80-mil thick high-density polyethylene (HDPE) liner to prevent the release of treated process water to the underlying soil and groundwater. A typical cross-section of the Effluent Turkey Nest is provided in Appendix 1, Figure 6.

The Effluent Pond receives neutralized process water from the Effluent Turkey Nest and serves as a storage vessel prior to reuse and/or discharge. The 10-foot deep pond is constructed to hold approximately 2,250,000 gallons of treated process water with a minimum freeboard of 2 feet. The Effluent Pond is lined with an 80-mil thick high density polyethylene (HDPE) liner to prevent the release of treated process water to the underlying soil and groundwater. A typical cross-section of the Effluent Pond is provided in Appendix 1, Figure 5. One pump at the Effluent Pond is used to reclaim treated water for reuse in the ZFP. Another pump is used to discharge treated effluent water through an HDPE discharge line (Outfall 001) to the same ditch that conveys storm water from the concrete level spreader installed within the bank of the Storm Water Pond (Outfall

002). The location of the discharge line and point are shown on Figure 2, Site Layout Map located in Appendix 1. The effluent discharge pump is rated for a maximum discharge of 140 gallons per minute. However, the average daily discharge rate is 29.4 gallons per minute. The maximum 24 hour discharge rate allowed by our permit is 41.6 gallons per minute.

#### 3.4 Sludge Dewatering

Approximately 3 gallons per minute of 30 percent solids from the Gypsum Thickener are bled from the Thickener Underflow Pump discharge into to the Vacuum Drum Filter. The 3-foot diameter by 3-foot long rotary drum filter dewaters the sludge to a minimum of 65 percent solids by weight. Filtrate is collected in a 100-gallon capacity Vacuum Receiver and pumped backed to the thickener center well. The gypsum filter cake is conveyed to a 160 square foot rectangular Storage Pad with 6-foot high concrete walls on three sides. A sump drain is used to carry any excess water or precipitation from the Storage Pad to the concrete Neutralization Area Sump located underneath the Gypsum Thickener. The dewatered gypsum cake is then hauled to mine site to be interred as part of Iluka's reclaim efforts.



# <u>Document 2:</u> NARRATIVE DESCRIPTION OF THE SITE

Iluka Resources' Processing site is situated on 36.6 acres in Sussex County near the town of Stony Creek (see Appendix 1, Figure 1).

Storm water drainage for 11.85 acres of the site, including the MSP building and associated facilities, is provided by Outfall 002 (see Appendix 1, Figure 2).

#### 1.0 Significant Materials

Significant materials used, proposed for use, or manufactured in the drainage area served by Outfall 002 include the following items:

- Raw mineral sand concentrate feed
- Off-road diesel fuel
- Dryer fuel oil
- Sulfuric acid
- The Effluent Treatment Plant (ETP)
  - Hydrated lime
  - Gypsum
- Kerosene
- Finished mineral sand products
- Tailings
- Small quantities of chemicals, lubricants and hydraulic oils

Each of these materials, except for the raw mineral sand concentrate feed, gypsum, and tailings are stored in enclosed containers or buildings, which prevent the contact of storm water with the material. Please refer to Appendix 1, Figure 2 for the locations of all items listed.

#### 2.0 Methods of Treatment, Storage, and Disposal

#### 2.1 Raw Mineral Sand Concentrate Feed

The mineral sand concentrate, which consists of approximately 90 percent mineral sands, is hauled by truck approximately 14 miles to the Mineral Separation Plant (MSP) for final separation and production of mineral sands products. After weigh-in on the scales located near the entrance to the site, the trucks dump the raw concentrate in an approximately 1.85-acre storage stockpile area located immediately north of the MSP.

Concentrate is removed from the stockpile and fed into the MSP's grizzly feed sump by the site's Volvo front end loader. The pollutant of concern associated with concentrate storage and plant feed is the concentrate material itself. The mineral sands in the stockpiles are naturally occurring and inert. None of the RCRA metals were observed in the results of Toxicity Characteristic Leaching Procedure (TCLP) testing that was performed on mineral sand concentrate and finished product material in 2000. The results of this testing can be found in Appendix 3, Item 3. Additional analytical results of samples taken from outfall 002 and required for the renewal of this permit are included in Appendix 3 as Item 4.

As seen in Table 1, the minerals have relatively high specific gravities ranging from 3.95 (leucoxene) to 4.75 (ilmenite). Therefore, it is unlikely that concentrate would be transported by surface water sheet flow resulting from small storm events. Storm water from the concentrate stockpile areas sheet flows until it intersects one of three unlined drainage channels that are located around the east, west, and south perimeter of the stockpile area. The drainage channels convey the storm water south to Retention Pond 3. In the event of a large storm event, minor amounts of concentrate could become available for transport to Retention Pond 3. If concentrate reaches the pond, it will settle out and Iluka will reclaim the valuable concentrate from the drainage channels and/or pond. Transport of mineral sands through outfall 002 from the Storm Water pond is highly unlikely.

Table 1: Properties of Minerals

| Property            | Ilmenite             | Rutile           | Leucoxene                             | Zircon             |
|---------------------|----------------------|------------------|---------------------------------------|--------------------|
| Chemical Formula    | FeTiO <sub>3</sub> , | Ti0 <sub>2</sub> | TiO <sub>2</sub> + FeTiO <sub>3</sub> | ZrSiO <sub>4</sub> |
| Specific Gravity    | 4.75                 | 4.22             | 3.95                                  | 4.69               |
| Magnetic Properties | Conductor            | Conductor        | Conductor                             | Non-Conductor      |
| magnetic Properties | Magnetic             | Non-Magnetic     | Weakly Magnetic                       | Non-Magnetic       |

#### 2.2 Off-Road Diesel Fuel

Iluka contracts dump trucks owned and operated by a third party to transport mineral sand concentrate to the MSP and tailings back to the mine site. These trucks are fueled and maintained off-site by the contractor. A 2,000-gallon above-ground storage tank (AST) that is located in the Tank Farm northwest of the MSP is used to store off-road fuel for Iluka's diesel powered equipment that includes the front-end loader used to load concentrate into the grizzly feed sump and the transfer pump located at the Storm Water pond. The 2,000-gallon AST is equipped with welded steel secondary containment and a rain shield.

#### 2.3 Dryer Fuel Oil

The primary pollutant of concern associated with feed drying activities is the dryer fuel (#2 fuel oil, #6 fuel oil, or on-specification used oil). Used oil for the primary dryer is obtained from off-site suppliers and meets Virginia Department of Environmental Quality's (VDEQ's) criteria for "on-spec" used oil as well has requirements set forth in the site's VDEQ Stationary Source Permit, Registration # 51220. Dryer fuel is stored in a 20,000-gallon AST that is located in the Tank Farm northwest of the MSP. The AST is equipped with welded steel secondary containment and a rain shield.

#### 2.4 Kerosene

Kerosene is burned in torpedo heaters which are used during the winter months to regulate the temperature of the plant. The kerosene is currently stored in one 200-gallon AST located to the south of the MSP. However, the tank is to be relocated to the Tank Farm northwest of the MSP in the near future.

#### 2.5 Sulfuric Acid

Sulfuric acid used in the Zircon Finishing Plant (ZFP, formerly Zircon Acid Leaching Circuit) is stored in a 5,000-gallon high density linear polyethylene (HDLPE) AST with concrete secondary containment located at the northwest corner of the MSP building.

#### 2.6 Effluent Treatment Plant

Acidic process water from the ZFP is neutralized in the Effluent Treatment Plant (ETP). The ETP consists of an enclosed steel silo which houses the Lime Storage Bin, the Lime Slurry Tank, and the Neutralization Tank; a Gypsum Mixing Tank; a Gypsum Thickener/Clarifier; a Filter Building; and a Filter Cake Storage Pad. Hydrated lime is loaded directly into the storage bin using a pneumatic conveyance system. All of the treatment plant equipment is built on concrete pads with provisions for drainage of washdown water, spills or leaks, and/or precipitation into a concrete Neutralization Area Sump located underneath the Gypsum Thickener. A sump pump conveys the collected water into the center well of the Gypsum Thickener and back into the treatment system. In the event of a pump or tank failure the water would likely be contained within the Neutralization Area Sump. If water escaped the ETP area it would flow east towards a ditch which drains into Retention Pond 3. A temporary berm could be constructed across the ditch by the on-site front-end loader to capture any release. Any sludge or precipitates contained in the process water would likely settle out before the catch basin due to their high specific gravities. All process water would be retained and pumped backed to the ETP.

The Effluent Turkey Nest stores neutralized effluent water from the ETP. The pond may also be used to temporarily store acidic effluent water in the event of a release inside the MSP. Any acidic water would be pumped to the ETP and neutralized. The 10-foot deep Effluent Turkey Nest is constructed with 1.5:1 horizontal to vertical (H:V) inside side slopes and bottom dimensions of 50-feet by 50-feet. The permitted minimum freeboard is 2 feet leaving 8 feet for storage of approximately 230,000 gallons of effluent water. The turkey nest is lined with an 80-mil thick high-density polyethylene (HDPE) liner to prevent the release of process water to the underlying soil and groundwater. The turkey nest is constructed in-ground; therefore, direct release to surface water is unlikely.

The Effluent Pond receives and stores neutralized effluent water from the Turkey Nest prior to reuse and/or discharge through outfall 001. The 10-foot deep pond is constructed with 4-foot high engineered berms with 2:1 (H:V) outside side slopes, 1.5:1 (H:V) inside slopes and bottom dimensions of 160-feet by 180 feet. The permitted minimum freeboard is 2 feet leaving 8 feet for storage of approximately 2,250,000 gallons of treated effluent water. The Effluent Pond is lined with an 80-mil thick HDPE liner to prevent the release of process water to the underlying soil and groundwater. Since 4 feet of the pond is constructed above ground, a direct release to surface water is possible in the event of a berm failure. Although the minimum freeboard is 2 feet, the normal operating freeboard is 3 feet to allow for storage capacity in the event of a release within the MSP and to limit the potential for berm failure. In the unlikely event of a berm failure, approximately 1 foot or roughly 275,000 gallons of water could be released from the pond, but would drain into Sediment Basin 2. A diesel transfer pump adjacent to the basin would be used to return the water to the ETP or Process Water Pond.

#### 2.7 Finished Mineral Sand Products

After mineral separation and concentration occurs and impurities are removed, the ilmenite and zircon products are temporarily stored in covered metal bins located on the east side of the MSP Building. Product is loaded into the bins by elevators.

Programmed logic controller (PLC) equipped loadout conveyors transfer the products to bulk railcars provided by CSX Railway. A weigh scale interlocked to a totalizer and the conveyor PLC unit stops the conveyor and closes the bin gate to the railcar when 100 tons has been transferred. Because the storage bins are covered, storm water does not to interact with the stored finished product.

#### 2.8 Tailings

Tailings are slurried and pumped to the Tails Stacker located west of the MSP. The tailings generally consist of quartz, staurolite and other minor sand-sized gangue minerals. These naturally occurring, inert materials do not pose a risk as pollutants to surface water. However, process and storm water drainage from the tailings is collected

and routed to the Tailings Turkey Nest where solids are settled out before pumping the water to Process Water Pond 1 for reuse. If initial zircon recoveries are not considered optimal, tailings may be reprocessed at the Mine Site Concentrator. To improve secondary zircon recovery from the tails, staurolite can be diverted from the Tails Stacker to the Staurolite Stacker located to the south of the MSP. Water from the stacker cyclone is directed to the Tailings Turkey Nest. Any additional drainage from the stacked staurolite flows to Retention Pond 3, where it is moved via the adjacent diesel transfer pump to Process Water Pond 2 for reuse. The stacked tailings are loaded into dump trucks and hauled back to the mine where they are backfilled into the mine pits and graded for reclamation.

#### 2.9 Small Quantities of Chemicals, Lubricants, and Hydraulic Oils

Minor amounts of various chemicals used in equipment maintenance and cleaning are stored in three primary locations within the MSP Building. Chemical storage in these enclosed areas does not pose a threat of release due to the minor quantities of materials stored. With the exception of the Volvo front end loader, all chemicals are used on equipment located within the MSP Building and the materials do not come into contact with storm water. A used and waste oil containment area is located on the paved area along the exterior wall of the MSP. Used and waste oil are stored in a sealed 200 gallon plastic collection drum. Used hydraulic oil, small amounts of waste oil, soiled absorbent pads and booms are stored in sealed 50 gallon metal drums or sealed 5 gallon plastic buckets. Storm water does currently come into contact with the collection vessels, however, as previously noted, these containers are sealed, and therefore storm water does not come into contact with the materials contained within. This area is inspected regularly by MSP and EHS personnel. Collection of the materials by either Safety-Kleen Systems or Siemens Water Technologies is scheduled as needed. In the unlikely event that one or more of the collection vessels is compromised, immediate efforts will be made to contain and recover the material on the paved surface. If initial containment efforts fail, the material would flow into the drainage ditch feeding into Retention Pond 3. Absorbent pads and booms would be deployed to arrest

the flow in the ditch. If any of the oil managed to make it to Retention Pond 3, absorbent pads and booms would be used to collect it. Any contaminated absorbent materials, water and/or soil resulting from such an event would be collected, treated and disposed of properly. Plans are currently underway to construct a metal shed which will shield this area from storm water.

#### 3.0 Past and Present Management Practices

Iluka has prepared a combined Storm Water Pollution Prevention and SPCC Plan to document procedures used to minimize the contact of materials with storm water runoff. This document is currently being updated. A copy of the SWPP/SPCC Plan is available for inspection in the Environmental, Health and Safety Office at the Administrative/Laboratory Building. As mentioned in the above section, chemicals are primarily stored in enclosed buildings or ASTs equipped with secondary containment. When this is not possible, the chemicals are stored in sealed containers and placed on paved surfaces in designated areas.

#### 4.0 Pesticide and Herbicide Use

Herbicides are not generally used at the site. A monthly service is used to control insects and rodents in the MSP and Administrative/Laboratory Building. A copy of the pest control agreement and MSDS sheets for the chemicals used on-site are included in Appendix 2 as Document 3.



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# Pest Control Service Agreement

#### HOUCHINS PEST CONTROL, INC.

2775 County Drive PETERSBURG, VA 23803 (804) 732-2930 Fax (804) 732-6842 1077

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BAYER CROP SCIENCE

P.O. Box 4913 Hawthorn Road Kansas City, MO 64120-0013

TRANSPORTATION EMERGENCY NON-TRANSPORTATION CALL CHEMTREC: 800-424-9300 BAYER EMERGENCY PHONE...: (800) 414-0244 INTERNATIONAL: 703-527-3887 BAYER INFORMATION PHONE.: (800) 842-8020 CHEMICAL PRODUCT IDENTIFICATION: PRODUCT NAME..... TEMPO SC Ultra Premise Spray PRODUCT CODE..... 21648 CHEMICAL FAMILY....: Pyrethroid Insecticide CHEMICAL NAME,....: Cyano(4-fluoro-3-phenoxyphenyl)methyl 3-(2,2 -dichloroethenyl)-2,2-dimethylcyclopropanecarboxylate SYNONYMS..... beta-cyfluthrin FORMULA..... C22 H18 C12 F N O3 COMPOSITION/INFORMATION ON INGREDIENTS: INGREDIENT NAME /CAS NUMBER EXPOSURE LIMITS CONCENTRATION (%) \*\*\*\*\* HAZARDOUS INGREDIENTS FCR 4545 Technical (beta-cyfluthrin) 68359-37-5 OSHA: Not Established 11.8 % ACGIH: Not Established

Specific chemical identity is withheld as a trade secret.

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ACGIH: Not Established

Product Code: 21648 Approval date: 05/06/2002

MSDS Page 1 Continued on next page

1-3 %

HAZARDS IDENTIFICATION: \*\*\*\*\*\*\*\*\*\*\*\*\*\* EMERGENCY OVERVIEW \* CAUTION! Color: Beige; Form: Liquid: Off-white to beige \* viscous liquid suspension; Odor: Chalky; Harmful if \* inhaled; Harmful if absorbed through skin; Causes eye \* irritation. POTENTIAL HEALTH EFFECTS: ROUTE(S) OF ENTRY..... Inhalation; Skin Contact; Skin Absorption; Eye Contact HUMAN EFFECTS AND SYMPTOMS OF OVEREXPOSURE: ACUTE EFFECTS OF EXPOSURE....: Skin and mucous membrane irritation may occur from contact with the product and produce symptoms such as itching, stinging, skin reddening or rash. Paresthesia (a tingling or burning sensation on the surface of the skin) may also result from skin contact. These are frequently reported symptoms associated with sufficient dermal exposure to alpha-cyano (Type II) synthetic pyrethroids and normally subside without treatment within 24 hours. The onset of these symptoms usually occurs 2-12 hours after exposure. The effects are temporary and are reversible. Based on the EPA Toxicity Category criteria, this material is mildly toxic by the oral and dermal routes of exposure. In addition, animal studies have shown that it can cause mild irritation to the conjunctiva of the eye with all irritation resolving within 7 days. CHRONIC EFFECTS OF EXPOSURE...: Based on animal studies, no adverse effects or symptoms would be expected from chronic exposure to this material. CARCINOGENICITY..... : This product is not listed by NTP, IARC or regulated as a carcinogen by OSHA. MEDICAL CONDITIONS AGGRAVATED BY EXPOSURE.....: No specific medical conditions are known which may be aggravated by exposure to this product. As with all materials which can cause upper respiratory tract irritation, persons with a history of asthma, emphysema, or hyperreactive airways disease may be more susceptible to overexposure. FIRST AID MEASURES:

FIRST AID FOR EYES.....: Hold eyelids open and flush with copious amounts of water for 15 minutes. Call a physician if irritation develops or persists

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MSDS Rage 2 Continued on next page

#### 4. FIRST AID MEASURES (Continued)

after flushing.

FIRST AID FOR SKIN....: Remove contaminated clothing immediately. Wash skin with soap and water, preferably preceded by a waterless hand cleaner. Get medical attention if irritation develops or persists. If signs of intoxication (poisoning) occur, get medical attention immediately.

FIRST AID FOR INHALATION: If a person is overcome by excessive exposures to aerosols or vapors of this material, remove to fresh air or uncontaminated area. If not breathing, give artificial respiration, preferably mouth-to-mouth. Get medical attention as soon as possible.

FIRST AID FOR INGESTION.: If ingestion is suspected, call a physician or poison control center. Drink one or two glasses of water and induce vomiting by touching back of throat with finger, or, if available, by administering syrup of ipecac. If syrup of ipecac is available, administer 1 tablespoonful (15 mL) of syrup of ipecac followed by 1 to 2 glasses of water. If vomiting does not occur within 20 minutes, repeat the dose once. Do not induce vomiting or give anything by mouth to an unconscious person.

NOTE TO PHYSICIAN.....: ANTIDOTE: No specific antidote is available. Treat victim symptomatically. Published data indicate vitamin E acetate can prevent and/or mitigate symptoms of paresthesia caused by synthetic pyrethroids. In case of poisoning, it is also requested that Bayer Corp., Agriculture Division, Kansas City, Missouri, be notified. Telephone: 1-800-414-0244

#### 5. FIRE FIGHTING MEASURES:

FLASH POINT.....: Greater than 200 F (93 C)

EXTINGUISHING MEDIA......: Water; Foam; Dry Chemical

SPECIAL FIRE FIGHTING PROCEDURES: Keep out of smoke. Cool exposed containers with water spray. Fight fire from upwind position. Use self-contained breathing equipment. Contain runoff to prevent entry into sewers or waterways. Equipment or materials involved in pesticide fires may become contaminated.

#### 6. ACCIDENTAL RELEASE MEASURES:

SPILL OR LEAK PROCEDURES.....: Isolate area and keep unauthorized people away. Do not walk through spilled material. Avoid breathing vapors and skin contact. Remove sources of ignition if combustible or flammable vapors may be present and ventilate area. Wear proper protective equipment. Dike contaminated area with absorbent granules, soil, sand, etc. If large spill, material should be recovered. Small spills can be absorbed with absorbent granules, spill control pads, or any absorbent material. Carefully sweep up absorbed spilled material. Place in covered container for reuse or disposal. Scrub contaminated area with soap and water. Use dry absorbent materials such as clay granules to absorb and

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## 6. ACCIDENTAL RELEASE MEASURES (Continued)

collect wash solution for proper disposal. Contaminated soil may have to be removed and disposed. Do not allow material to enter streams, sewers, or other waterways or contact vegetation.

HANDLING AND STORAGE: STORAGE TEMPERATURE(MIN/MAX): 0 F/30 day avg. not to exceed 100 F SHELF LIFE...... Time/temperature-dependent. Contact Bayer for specific information. SPECIAL SENSITIVITY..... Not cstablished HANDLING/STORAGE PRECAUTIONS: Store in a cool, dry area designated specifically for pesticides. PERSONAL PROTECTION: EXE PROTECTION REQUIREMENTS...... Goggles should be used to prevent liquid from getting into eyes. SKIN PROTECTION REQUIREMENTS...... Avoid skin contact. Wear long sleeves and trousers. HAND PROTECTION REQUIREMENTS.....: Chemical-resistant gloves such as neoprene VENTILATION REQUIREMENTS..... Control exposure levels through the use of general and local exhaust ventilation. RESPIRATOR REQUIREMENTS...... When respiratory protection is necessary under the conditions of use, wear a NIOSH-approved organic vapor respirator with dust and mist filter. ADDITIONAL PROTECTIVE MEASURES ....: Clean water and soap should be available for washing in case of eye or skin contamination. Waterless hand cleaner use is often more effective than soap and water. Sensitive areas of the skin and mucous membranes can become contaminated indirectly. Educate and train employees in safe use of the product. Follow all label instructions. Launder clothing separately after use. Wash thoroughly after handling. PHYSICAL AND CHEMICAL PROPERTIES: PHYSICAL FORM..... Liquid APPEARANCE..... Off-white to beige viscous liquid suspension COLOR..... Beige ODOR..... Chalky MOLECULAR WEIGHT..... 434.3 (for beta-cyfluthrin) pH ....: 7-8 BOILING POINT ..... Not established MELTING/FREEZING POINT...: Approx. 20 F

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#### 9. PHYSICAL AND CHEMICAL PROPERTIES (Continued)

VISCOSITY....: 1060 cps @ 23 C

SOLUBILITY IN WATER : Not established

SPECIFIC GRAVITY : 1.06 @ 20 C/20 C

BULK DENSITY...: Not established

VAPOR PRESSURE : 7.2 x 10-9 mm Hg @ 20 C (for beta-cyfluthrin)

a so so min ing a so o (row beca-cyriuchrin)

#### 10. STABILITY AND REACTIVITY:

STABILITY..... This is a stable material.

HAZARDOUS POLYMERIZATION ...: Will not occur.

INCOMPATIBILITIES..... Alkaline media; reacts with methanol; incompatible

with many disinfectants.

INSTABILITY CONDITIONS....: Not established DECOMPOSITION PRODUCTS....: Not established

#### 11. TOXICOLOGICAL INFORMATION:

Only acute studies have been performed on this product as formulated. The non-acute information pertains to the active ingredient, cyfluthrin technical, and its enriched isomer mixture, BAY FCR 4545 technical.

#### ACUTE TOXICITY

ORAL LD50.....: Male rat: 960 mg/kg -- Female rat: 1150 mg/kg DERMAL LD50.....: Male and Female Rat: >2000 mg/kg

INHALATION LC50...: 4 hr exposure to Liquid Aerosol: Male and Female Rat: >1.72 mg/L (analtyical) -- 1 hr exposure to Liquid Aerosol (extrapolated from 4 hr): Male and Female Rat: >6.88 mg/L (analytical)

EYE EFFECTS.....: Rabbit: Mild irritation to the conjunctiva was observed with all irritation cleared within 7 days post-treatment.

SKIN EFFECTS.....: Rabbit: Not a dermal irritant

SENSITIZATION.....: Guinea pig: Not a dermal sensitizer
SUBCHRONIC TOXICITY...: FCR 4545: In a 13 week dog study, FCR 4545 was
administered at dictary concentrations of 10, 60 or 360 ppm. Effects included
vomiting and diarrhea after feeding, decreased body weight gain, and motor
disturbances in the hind limbs. The no-observed effect-level (NOEL) was 60
ppm. In a 13 week study using rats, FCR 4545 was administered at dietary
concentrations of 30, 125 or 500 ppm. Effects included reduced body weight
gains and feed consumption, uncoordinated gait, and skin injuries of the neck
and head from excessive preening due to the local irritant effect of the test
material. The NOEL was 125 ppm. In a 4 week inhalation study, rats were
exposed to FCR 4545 at liquid aerosol concentrations of 0.2, 2.7 or 23.5
mg/m3. Effects observed included ungroomed fur, piloerection, hyper- and
hypoactivity, reduced body weight gains, reduced organ weights (thymus and
spleen), and hematological changes. The NOEL was 0.2 mg/m3 based on decreased
body weight gains. CYFLUTHRIN: In a 3 week dermal toxicity study, cyfluthrin

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## 11. TOXICOLOGICAL INFORMATION (Continued)

was administered to rats for 6 hours/day at dose levels of 100, 340 or 1000 mg/kg. Animals received a total of 17-18 applications in a period of 22-23 days. An additional control and high-dose group were treated and maintained for 14-15 days following treatment so as to ascertain the extent of recovery. Effects observed included reduced feed consumption, red nasal discharge, urine stains, and findings at the dose site (scabbing, crusty, discolored and raised zones). Histologically, epidermal and dermal alterations in treated skin were observed in animals of the mid- and high-dose groups. Similar, but slightly less severe microscopic alterations were also observed in the high-dose recovery group. The overall NOEL was 100 mg/kg. In a 13 week inhalation study, rats were exposed to cyfluthrin at acrosol concentrations of 0.09, 0.71 or 4.51 mg/m3 for 6 hours/day, 5 days/week. The NOEL was 0.09 mg/m3 based on reduced body weight gains.

CHRONIC TOXICITY....: Cyfluthrin has been investigated in chronic feeding studies using two different strains of rats. In each study, cyfluthrin was administered for 2 years at dietary concentrations ranging from 50 to 450 ppm. Body weight gains were decreased at concentrations of 150 ppm and greater. Changes in clinical chemistries occurred at 450 ppm. In one of the studies, histopathology revealed a numerical increase in mammary gland adenocarcinomas at 450 ppm. This finding was not statistically significant when compared to the controls and is not considered to be compound-related. In each study, the overall NOEL was 50 ppm based on decreased body weight gains. In a 1 year feeding study, dogs were administered cyfluthrin at dietary concentrations of 50, 100, 360 or 650 ppm. Beginning on week 8, the high dose was reduced to 500 ppm for the remainder of the study due to severe clinical neurological symptoms. Body weights were decreased for animals of the high-dose. Neurological findings (gait abnormalities and postural reaction deficits) were observed at doses of 360 and greater. The NOEL was 100 ppm.

CARCINOGENICITY.....: Cyfluthrin was investigated for carcinogenicity in chronic studies using rats and mice at maximum levels of 450 and 800 ppm, respectively. There was no evidence of a carcinogenic potential observed in either species.

MUTAGENICITY.....: In vitro and in vivo mutagenicity studies have been conducted on BAY FCR 4545 technical, all of which are negative. Numerous in vitro and in vivo mutagenicity studies have been conducted on cyfluthrin, all of which are negative.

DEVELOPMENTAL TOXICITY: FCR 4545: In a developmental toxicity study, Bay FCR 4545 technical was administered orally to rats during gestation at doses of 3, 10 or 40 mg/kg. At the lethal and maternally toxic dose of 40 mg/kg, there was a decrease in fetal body weights and an increased incidence of skeletal findings. The NOELs for maternal and developmental toxicity were 3 and 10 mg/kg, respectively. Cyfluthrin: In developmental toxicity studies using rats, cyfluthrin was administered during gestation by oral gavage at doses ranging from 1 to 30 mg/kg. The overall NOEL from these studies for maternal toxicity was 3 mg/kg. No developmental effects were observed at any of the doses tested. In each study, the NOEL for developmental toxicity was equivalent to the highest dose tested. The NOELs for developmental toxicity for the initial study and the subsequent study were 30 and 10 mg/kg, respectively. Rabbits were administered cyfluthrin during gestation by oral gavage at doses ranging from 5 to 180 mg/kg. At maternally toxic levels, there was an increased incidence of post-implantation losses. The overall

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#### 11. TOXICOLOGICAL INFORMATION (Continued)

NOEL derived from these studies for both maternal and developmental toxicity was 20 mg/kg. In an inhalation study, rats were exposed during gestation to cyfluthrin at aerosol concentrations of 0.46, 2.55 or 11.9 mg/m3 for 6 hours/day. NOELs for maternal and developmental toxicity were less than 0.46 and 0.46 mg/m3, respectively.

REPRODUCTION.....: In a reproduction study, cyfluthrin was administered to rats for 3 generations at dietary concentrations of 50, 150 and 450 ppm. Reproductive effects observed at parentally toxic levels included reductions in viability, lactation, litter size, feed consumption, and pup birth weights and body weight gains. Coarse tremors were observed in some offspring at 450 ppm. The NOEL for both parental and reproductive effects was 50 ppm. In another reproduction study, cyfluthrin was administered to rats for 2 generations at dictary concentrations of 50, 125 or 400 ppm. Coarse tremors occurring in conjunction with parental Loxicity were observed in the offspring in the mid- and high-dose groups. Based on this finding, the neonatal NOEL was 50 ppm. The NOELs for parental and reproductive toxicity were 50 and 400 ppm, respectively.

NEUROTOXICITY .....: Numerous neurotoxicity studies have been conducted on cyfluthrin. Oral gavage studies using hens have indicated that at extremely high dosc levels (5000 mg/kg), minimal nerve damage occurs. When rats were administered cyfluthrin daily at oral doscs of 40 to 80 mg/kg for 14 days, minimal nerve effects were seen. These effects were completely reversible within a 3 month recovery period. In dermal and inhalation studies which are relevant to field exposure, there was no evidence of delayed neurotoxicity in hens. In a special investigative study, litters of meonatal mice (10 days of age) and their mothers were exposed to aerosol concentrations of 5, 15, or 50 mg/m3 for 6.3 hours/day for 7 successive days. Motor activity was measured in the offspring at 4 months of age (approximately 3.5 months post-exposure). At 50 mg/m3, all of the offsprings died or were sacrificed in a moribund state following the first exposure. Mortalities were not observed at any of the other levels. Clinical symptoms were observed immediately after exposure in young mice at 15 mg/m3, and included decreased motility, temporary scratching, and tonic convulsions. There was an increase in motor activity in mice at 15 mg/m3. Histopathological investigations did not reveal any treatment-related findings in mice at the age of 4 months.

#### 12. ECOLOGICAL INFORMATION:

This product is extremely toxic to fish and aquatic invertebrates, and is highly toxic to bees. Bayer will provide a summary of specific data upon written request. As with any pesticide, this product should be used according to label directions and should be kept out of streams, lakes and other aquatic habitats of concern. In event of a spill emergency, call 1-800-414-0244.

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| 13. DISPOSAL CONSIDERATIONS  |
|--|
| WASTE DISPOSAL METHOD: Follow container label instructions for disposal of wastes generated during use in compliance with the FIFRA product label. In other situations, bury in an EPA approved landfill or burn in an incincrator approved for pesticide destruction.  EMPTY CONTAINER PRECAUTIONS.: Do not reuse container without written permission and instructions from Bayer. Empty, clean and dispose in accordance with state and local laws. |
| 14. TRANSPORTATION INFORMATION:  |
| TECHNICAL SHIPPING NAME : beta-Cyfluthrin  FREIGHT CLASS PACKAGE : Insecticides, NOI, NMFC 102100  PRODUCT LABEL : TEMPO SC Ultra Premise Spray  |
| DOT (DOMESTIC SURFACE)   |
| HAZARD CLASS OR DIVISION: Non-Regulated  |
| It is not expected that a mist or vapor hazard would exist from the normal transportation of this liquid substance.  |
| IMO / IMDG CODE (OCEAN)  |
| HAZARD CLASS DIVISION NUMBER: Non-Regulated  |
| ICAO / IATA (AIR)  |
| HAZARD CLASS DIVISION NUMBER: Non-Regulated  |
|  |
| 15. REGULATORY INFORMATION:  |
| OSHA STATUS This product is hazardous under the criteria of the Federal OSHA Hazard Communication Standard 29  |
| FSCA STATUS: This product is exempt from TSCA Regulation under FIFRA Section 3 (2)(B)(ii) when used as a   |
| pesticide.<br>CERCLA REPORTABLE QUANTITY: No components listed.  |
|  |

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#### 15. REGULATORY INFORMATION (Continued)

SARA TITLE III:

SECTION 302 EXTREMELY

HAZARDOUS SUBSTANCES..: No components listed.

SECTION 311/312

HAZARD CATEGORIES.....: Immediate Health Hazard

SECTION 313

TOXIC CHEMICALS.....: beta-Cyfluthrin (11.8%) - CAS # 68359-37-5

RCRA STATUS...... If discarded in its purchased form, this product would not be a hazardous waste either by listing or by characteristic. However, under RCRA, it is the responsibility of the product user to determine at the time of disposal, whether a material containing the product or derived from the product should be classified as a hazardous waste. (40 CFR 261.20-24)

16. OTHER INFORMATION:

NFPA 704M RATINGS:

Health Flammability Reactivity Other

1 1

0-Insignificant 1=Slight 2=Moderate 3=High 4=Extreme

Bayer's method of hazard communication is comprised of Product Labels and Material Safety Data Sheets. NFPA ratings are provided by Bayer as a customer service.

REASON FOR ISSUE..... Create new MSDS

PREPARED BY..... V. C. Standart APPROVED BY...... D. C. Eberhart

TITLE..... & Stewardship

SUPERSEDES DATE..... Nonc MSDS NUMBER..... 29752

This information is furnished without warranty, expressed or implied, except that it is accurate to the best knowledge of Bayer Corporation. The data on this sheet relates only to the specific material designated herein. Bayer Corporation assumes no legal responsibility for use or reliance upon these data.

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It's Really Going Places.

#### Consumer Product Information

#### TalstarOne™ Multi-Insecticide

Date Prepared: 23 April, 2004

#### 1. Product Information

Dilution of TalstarOne multi-insecticide, EPA Reg. # 279-3206 Chemical Family: Pyrethraid Insecticide

#### 2. Manufacturer Information

#### Manufacturer:

FMC Corporation Agricultural Products Group 1735 Market Street Philadelphia, PA 19103

#### Telephone Numbers:

Emergency Phone: (800)-331-3148 Spill Emergency: (800)-331-3148 Technical Information: (800)-321-1362

#### 3. Active Ingredient

CAS No.

Bifenthrin (0.12%) dilution Typical use rates are between 0.001% and 0.06% 82657-04-3

#### 4. General Information and Precautions

Highly toxic to fish and aquatic organisms. Care should be used when spraying to avoid fish and reptile pets. Wash thoroughly with soap and water after handling.

#### 5. Health Hazard Information

General Health/Toxicity Information for 0.12% Suspension:
Acute Oral LD50 >20,000 mg/kg
Acute Dermal LD50 >10,000 mg/kg

This diluted material has a low oral and dermal toxicity, it is minimally irritating to the eyes and non-irritating to the skin. In rare occurences, exposure to TalstarOne multi-insecticide end use dilution produces skin sensations such as burning, numbness or tingling during application. These sensations generally occur in the area of the body contacted by the diluted product and last for a few hours. This reaction, should it occur, produces some discomfort but does not result in skin damage.

#### 6. First Aid Procedures (Dilutions or Emulsions Only)

- Ingestion: Drink 1 or 2 glasses of water. Never give anything by mouth to an unconscious person. If any discomfort persists, obtain medical attention.
- Inhalation: Remove to fresh air. If breathing difficulty or discomfort occurs and persists, obtain medical attention.

Dermal: Wash with plenty of soap and water

· Eye: Flush with plenty of water. Get medical attention if irritation occurs and persists

Note to Physician or Emergency Personnel: TalstarOne multi-insecticide suspension contains a low concentration
of the pyrethroid bifenthrin and primarily water. Should reversible skin sensations occur, skin salves have been
found useful in reducing discomfort. Treatment is otherwise removal of exposure followed by symptomatic and
supportive care.

#### 7. Fire and Hazards Information

• The water-based suspension of TalstarOne multi-insecticide is not explosive or flammable.

#### 8. Environmental Hazards Information

• This product is extremely toxic to fish. Do not contaminate water when disposing of equipment wash-waters. Do not apply directly to any body of water. Care should be used when spraying to avoid fish and reptiles.

#### 9. Storage and Disposal and Spill Information

- Keep out of reach of children. Store in a cool, dry place and avoid excess heat. Do not put concentrate or dilute material into food or drink containers.
- Isolate and post spill area. Keep animals and unprotected persons out of area. Keep emulsion out of streams
  and sewers. Dike to confine spills, and absorb with absorbent such as clay, sand or cat litter. Place in a DOT
  approved drum and contact appropriate regulatory agencies prior to disposal. To decontaminate spill area,
  tools and equipment, wash with a detergent/water mixture and properly dispose of solution as contaminated
  waste.

#### 10. Additional Information

 For safety and health information concerning the undiluted concentrate product, refer to the Material Safety Data Sheet for TalstarOne multi-insecticide.

Disclaimer: The information and recommendations contained herein are based upon data believed to be correct. However, no guarantee or warranty of any kind, expressed or implied, is made with respect to the information contained herein.

Status: Effective 4/2004
TalstarOne and FMC Logo - FMC trademarks
B0039

## CONTRAC®

MSDS Date of Issue: FEBRUARY 1995

|  | 100011 111 00104   |   | R (608) 241-0:   | 202                          | Contact<br>Poison (  | Control Center                         |
|--|--|---|--|------------------------------|--|--|
| Coumarin CHEMICAL FORMULA: C30H23BrO4 SI   |  | VJD/CA  |  | 1.7                          | Poison (<br>CAS NO.:<br>28772-56-  |  |
| C30H23BrO4   |  | 3-{3-(4'-   | AL NAME & SYNONYMS<br>-Bromo-[1,1'-biphenyl]<br>oxy-2H-1-benzopy   |                              |  |  |
| Si   |  | 4-hydr  | NAME & SYNONYMS:   | ran-2-one                    |  |  |
|  | ECTION 1. H  | AZARD   | OUS INGREDI  | ENTS                         |  |  |
| ACTIVE INGREDIENTS:  | LCTION I. II   | ALAND   | OUS LIGREDI  |                              | , T  | CURRENT TLV                            |
| Bromadiolone   |  |   |  | 0.000                        | 005  | 500 mg/kg                              |
|  |  |   |  |                              |  | based on<br>beagle dog                 |
|  |  |   |  |                              |  |  |
|  |  |   |  |                              |  |  |
| SECTIO   | N II. PHYSIC   | AL DAT  | A OF ACTIVE  | INCRE                        | DIENT  | ,                                      |
| APPEARANCE & ODOR:   | MOLECULAR WEIG   |   | MELT POINT:  |                              |  | GRAVITY:                               |
| White odorless powder  | 527.4<br>COLOR:  | _   | 200-210°C  |                              | BOILING  | NA<br>POINT:                           |
| NA<br>VAPOR PRESSURE:  | White<br>SOLUBILITY:   |   | 0.33 gm/co   | 2                            |  | NA<br>TION RATE:                       |
| NA   | 12 mg/l (water   |   | NA   |                              | _  | NA NA                                  |
|  | N III. FIRE &  | & EXPLO   | OSION DATA   | OF PRO                       | DUCT   |  |
| FLASH POINT F (METHOD USED):<br>NA   |  | FL  | AMMABLE LIMIT:<br>NA   |                              | AUTOI  | GNITION TEMP<br>NA                     |
| EXTINGUISHING MEDIA:   | th water form  | innet non   |  |                              | 1  | 1363                                   |
| SPECIAL FIRE FIGHTING PROCE  | th water, foam or<br>EDURES:   | men gas.  |  |                              |  |  |
| None<br>UNUSUAL FIRE OR EXPLOSION  | HAZARDS:   |   |  |                              |  |  |
| None   |  |   |  |                              |  |  |
| SECTION IV. R  |  |   | DATA OF AC   | TIVE I                       | NGREI  | DIENT                                  |
| Stable   | NA NA  |   |  |                              |  |  |
| POLYMERIZATION:<br>Will not occur.   | CONDITIONS TO A  | VOID:   |  |                              |  |  |
| NCOMPATABILITY (MATERIALS<br>Strong Bases  |  | 1   | HAZARDOUS DECOMI<br>Carbon Monoxie   |                              |  |  |
| onong mases  | SECTION  | V. TO   |  | ue/ Caroo                    | III DIOXIO   | ic                                     |
| .050,ORAL (INGESTION)<br>1.125 mg/kg [Rat]<br>1.75 mg/kg [Mouse  |  | AL (SKIN CO   | NTACT): (1% Al) IN   | HALATION:                    | LC50   | (1% A                                  |
| ISH LCSO:  | 11 11 11 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1   | g/kg [Rabb  |  | 9.23 mg/1                    | x 1 hr   | [Rat]                                  |
| 1.4 mg/l [Rainbow Trou<br>3.0 mg/l [Bluegill sun   | it] Non-i  | EYE IRRITA<br>rritating   | TION: (1% Ab Li  | 050 ORAL:<br>10 mg/kg        | [Dog]  | CTECH                                  |
| Eyes: Flush with plenty of v<br>Ingestion: Administer Vit  | water. Skin; W<br>tamin K   intrarm<br>peat as necessary   | uscularly   | or orally as indicate  | alation:<br>ed in bish       | ydroxyco   |  |
|  | MATION:  |   | monitoring of proti  | hrombin t                    | imes.  | oumarin                                |
| PECIAL PROTECTION INFOR  | MATION:  |   | monitoring of proti  | hrombin t                    | imes.  | oumarin                                |
| None SECTION VII. CO   |  | PROTEC  |  | hrombin t                    | imes.  |  |
| SECTION VII. CO  |  | PROTEC  |  | hrombin t                    | imes.  |  |
| SECTION VII. CO ESPIRATOR TYPE. None  None   | ONTROL & P   |   | TIVE MEASUR  | RES OF                       | imes.  |  |
| SECTION VII. CO  SPIRATOR TYPE  None  None  None  None  None  None  None  None  None   | ONTROL & P   |   | TIVE MEASUR  | tES OF                       | imes.  |  |
| SECTION VII. CO SECTION VII. CO ESPIRATOR TYPE: None YE PROTECTION: None None None None None None None None  | ONTROL & P   | bber gloves   | FIVE MEASUR  | RES OF                       | PROD   | UCT                                    |
| SECTION VII. CO  SECTION VII. CO  ESPIRATOR TYPE: None  None None None SECTION VII. CO  SECTION VII. CO  | ONTROL & P   | bber gloves   | TIVE MEASUR  | RES OF                       | PROD   | UCT                                    |
| SECTION VII. CO  SECTION VII. CO  ESPIRATOR TYPE: None  None None THER PROTECTIVE MEASURES: None SECTION VII.  SECTION VII. CO   | ONTROL & P   | bber gloves   | FIVE MEASUR  | RES OF                       | PROD   | UCT                                    |
| SECTION VII. CO SECTION VII. CO ESPIRATOR TYPE None None THER PROTECTIVE MEASURES: None SECTION VI TEPS TO BE TAKEN IN CASE MAI General clean-up.  | ONTROL & P   | bber gloves   | FIVE MEASUR  | RES OF                       | PROD   | UCT                                    |
| SECTION VII. CO  SECTION VII. CO  ESPIRATOR TYPE: None  None  None  SECTION VII  SECTION VII  AND PROTECTIVE MEASURES: NONE  SECTION VII  SECTION VI | ONTROL & P GLOVES Rut  VIII. SPILL O   | PR LEAK   | VEN N  | EES OF                       | PROD   | UCT                                    |
| SECTION VII. CO SPIRATOR TYPE None None None SECTION VII. CO SEPPRATOR TYPE None None SECTION VII SEPROTECTIVE MEASURES: None SECTION VII  | ONTROL & P GLOVES Rul  VIII. SPILL O GENTAL IS WELEASED (  | PR LEAK   | FIVE MEASUR  VENI  N  X PROCEDURE  | RES OF                       | PROD  RODUC  | T plicable Feder                       |
| SECTION VII. CO SETTION VII. CO SEPTIMATOR TYPE. None None None SECTION VII. CO SECTION VII. C | ONTROL & P GLOVES Rul  VIII. SPILL O FERIAL IS RELEASED (  according to label  CIAL PRECAL   | PR LEAK OR SPILLED:   | FIVE MEASUR  VENI  N  PROCEDURE  15, must be disposed  & STORAGE   | RES OF                       | PROD  RODUC  | T T plicable Feder                     |
| SECTION VII. CO  SECTION VII. CO  ESPIRATOR TYPE  None  None  SECTION VII.  SEPROTECTION:  None  SECTION VII.  SECTION VIII.  SECTION VIII. | ONTROL & P  GLOVES. Rut  VIII. SPILL O  GENERAL IS RELEASED of according to label  CIAL PRECAI   | PR LEAK OR SPILLED:  I instruction UTIONS CRAGE SHELF             | FIVE MEASUR  VENI  N  PROCEDURE  15, must be disposed  & STORAGE   | DATA (                       | PRODUC   | T  DDUCT                               |
| SECTION VII. CO ESPIRATOR TYPE. None  None  SECTION VII. CO ESPIRATOR TYPE. None None SECTION VII. SECTION VIII. SECTION VII. SECTION V | GLOVES Rul  GLOVES Rul  VIII. SPILL O  GENERAL IS RELEASED of according to label  CIAL PRECAI  C. AVE  T. MOISTURE:  | DR LEAK OR SPILLED:  UTIONS CRAGE SHELF Bait is sta               | FIVE MEASUR  VENT N  C PROCEDURE  IS, must be disposed  & STORAGE  LIFE  | DATA (                       | PRODUC   | T  DDUCT                               |
| SECTION VII. CO ESPIRATOR TYPE None None None None SECTION VII. CO ESPIRATOR TYPE None None SECTION OF SECTION | ONTROL & P  GLOVES Rul  GLOVES | PR LEAK OR SPILLED:  I instruction UTIONS CRAGE SHELF Bait is sta | FIVE MEASUR  VENT N  C PROCEDURE  IS, must be disposed  & STORAGE  LIFE  | DATA (                       | PRODUC   | T  DDUCT                               |
| SECTION VII. CO ESPIRATOR TYPE. None  SECTION VII. CO ESPIRATOR TYPE. None None SECTION VII. SECTION VII. None SECTION VII. SECTION VIII. SECTION VII. SECTION VII. SECTION VII. SECTION VII. SECTION VII. SECTION VIII. SECTI | GLOVES RULE  GLOVES RULE  VIII. SPILL O  GENERAL IS RELEASED of label  CIAL PRECAL  AVE  E. MOISTURE:  Hight and humidity  SOLING AND STORING.  ms or ponds.   | PR LEAK OR SPILLED:  I instruction UTIONS ORAGE SHELF Bait is sta | FIVE MEASUR  VENT N  C PROCEDURE  IS, must be disposed  & STORAGE  LIFE  | DATA ( of 1 year             | PRODUC   | T  DDUCT                               |
| SECTION VII. CO ESPIRATOR TYPE None  None  SECTION VII. CO ESPIRATOR TYPE None  None  SECTION IN HER PROTECTIVE MEASURES. None  SECTION IN HER PROTECTIVE MEASURES. NONE  SECTION IN HER PROTECTIVE MEASURES. NONE  SECTION IN ASTE DISPOSAL METHOD: Product that cannot be used State or Local procedures. ROOM temperature EROAM TEMPERATURE: ROOM temperature ERICAL SENSITIVITY (HEAT, LIGHT AVOID deposure to I HECALTIONS TO BE TAKEN IN IN AVOID LIGHT SENSITIVITY (HEAT, LIGHT AVOID LIGHT SENSITIVI | GLOVES RULE  GLOVES RULE  VIII. SPILL O  GENERAL IS RELEASED of label  CIAL PRECAL  AVE  E. MOISTURE:  Hight and humidity  SOLING AND STORING.  ms or ponds.   | PR LEAK OR SPILLED  I instruction UTIONS PRACE SHELF Bait is sta  | FIVE MEASUR  VEN.  N  S PROCEDURE  IS, must be disposed  & STORAGE  LIFE.  ble for a minimum  ATA OF PROL  INICAL SHIPPING NAM                 | DUCT                         | PROD  RODUC  The property of t | T  plicable Feder  DDUCT  ored properl |
| SECTION VII. CO SECTION VII. CO SESTIMATOR TYPE. None None SECTION VII. CO SESTIMATOR TYPE. None SECTION VII. SECTION VIII. SECTION VII. SECTION VIII. SE | ONTROL & P  GLOVES Rut  VIII. SPILL O  GERIAL IS WELEASED (  AVE  E.  H. MOISTURE:  Hight and hurndity  Hight and hurndity  MITTON X. SHIP   | PR LEAK OR SPILLED  I instruction UTIONS PRACE SHELF Bait is sta  | FIVE MEASUR  VENI  N  PROCEDURE  is, must be disposed  & STORAGE  LUTE  ble for a minimum  | DUCT                         | PROD  RODUC  The property of t | T  plicable Feder  DDUCT  ored properl |
| SECTION VII. CO  ESPIRATOR TYPE.  None  None  None  None  None  None  SECTION VII. CO  ESPIRATOR TYPE.  None  SECTION VII.  SECT | ONTROL & P  GLOVES Rut  VIII. SPILL O  GERIAL IS WELEASED (  AVE  E.  H. MOISTURE:  Hight and hurndity  Hight and hurndity  MITTON X. SHIP   | PPING D   | FIVE MEASUR  VENT N  S PROCEDURE  IS, must be disposed  & STORAGE  LIFE LIFE LIFE LIFE LIFE ATA OF PROL  INICAL SHIPPING NAN Rodenticide cont. | of accord  DATA (  of 1 year | PROD  RODUC  The property of t | T  plicable Feder  DDUCT  ored properl |
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# APPENDIX 3 LAB REPORTS

ITEM 1

## RECEIVED

SEP 19 2006

## PRO



September 18, 2006

Virginia Operations 12472 St. John Church Rd. Stony Creek, VA 23882

Iluka Resources Inc.

Hand Delivered

T: +1 434 348 4300 F: +1 434 246 3039

www.iluka.com

RE:

Report of Analysis: Whole Effluent Toxicity (WET) conducted by Coastal Bioanalyst Inc. on Treated Effluent from Outfall 001 collected July 26, 2006 at the Iluka Resources Inc. Old Hickory Processing Plant

VPDES Permit # VA0090981

3rd Quarter 2006

Mr. Corwin Chamberlain Environmental Specialist II Department of Environmental Quality - Piedmont Regional Office 4949-A Cox Road Glen Allen, VA 23060

Dear Mr. Chamberlain:

The 3rd Quarter 2006 Whole Effluent Toxicity (WET) sampling was conducted on July 26, 2006. Representatives of the Iluka Resources Inc. Environmental, Health and Safety Department and P. D. & J. Licensed Environmental Consultants conducted the sampling and Coastal Bioanalyst Inc. of Gloucester, VA conducted the analyses. Results of the Whole Effluent Toxicity sampling are attached. If you have any questions or require additional information please contact me at 434.348.4316 Direct or 804.721.7312 mobile.

Regards,

Kevin Rideout EHS&T Technician

Attachments:

1) Results of Whole Effluent Toxicity (WET) sampling conducted July 26, 2006 - 12 letter size pages – 2 copies

Cc: Ray R. Jenkins, Jr., Environmental Engineer Senior, DEQ-PRO Shawn Conaway, Technical Manager, Iluka Resources Inc. Jack Rayburn, EHS &T Supervisor, Iluka Resources Inc.

Client: Iluka Resources, Inc. Project ID: ILUK0602 Client Sample ID: Outfall 001 Permit No: VA0090981 Sample Period: 7/26/06



## Report of Analysis: Whole Effluent Toxicity (WET)

| Submitted To: Mr. Kevin Rideout Iluka Resources, Inc. 12472 St. John Church Road Stony Creek, VA 23882-0126 | Prepared By: Coastal Bioanalysts, Inc. 6400 Enterprise Court Gloucester, VA 23061 (804) 694-8285 www.coastalbio.com Contact: Peter F. De Lisle, Technical Director |
|---|--|
|---|--|

| Species-Test Method 48-h L/C50 95% C.L. |        |       |
|---|--------|-------|
|   | 1 7.11 | MOARC |
| C. dubia EPA 2002.0 56.8 44.8-76.0      | 1.76   | NOAEC |
| P. promelas EPA 2000.0 >100 N/A         | <1.00  | N/A   |

Note: Details regarding test conduct and data analysis provided in attached bench sheets and printouts as applicable.

| Acute Test Biological Summary Data Species-Method Endpoint |               |         |     | Sample Concentration (%) |    |          |          |
|--|---------------|---------|-----|--------------------------|----|----------|----------|
|  | Endpoint      | Control | 6.3 | 13                       | 25 | 50       | 100      |
| C. dubia EPA 2002.0  | Survival (%): | 100     | 100 | 100                      | 85 | 60       | -        |
| P. promelas EPA 2000.0                                     | Survival (%): | 100     | 100 | 100                      | 05 | 60<br>90 | 20<br>80 |

| Species-Method         | End Date/Time                | Organism<br>Source | Hatch/Harvest<br>Date/Time   | Acclimation<br>Temp. | Acclimation<br>Water | Test<br>Aerated? |
|------------------------|------------------------------|--------------------|------------------------------|----------------------|----------------------|------------------|
| C. dubia<br>EPA 2002.0 | 7/26/06 1525<br>7/28/06 1515 | CBI ·              | 7/25/06 1615                 | 1                    | Mod. Hard            | Aerateu:         |
| P. promelas            | 7/26/06 1520                 | Stock<br>CBI       | 7/26/06 1010<br>7/20/06 1100 | 25° C                | Syn. FW              | No               |
| EPA 2000.0             | 7/28/06 1510                 | Stock              | 7/21/06 1100                 | 25° C                | Mod. Hard<br>Syn. FW | No               |

| Sample/Dilution Water Data                  | Acut                              | e Test         |
|---|-----------------------------------|----------------|
| Water Quality Parameter (Units)             | Sample                            | Dilution Water |
| Arrival Temperature (°C)                    | 4                                 | N/A            |
| Use Temperature (°C)                        | 25                                | 25             |
| Conductivity (µS/cm)                        | 2670                              | 291            |
| pH (S.U.)                                   | 8.02                              | 7.68           |
| Dissolved Oxygen (mg/l)                     | 8.2                               | 8.2            |
| Total Hardness (mg/l as CaCO <sub>3</sub> ) | 1686                              | 84             |
| Alkalinity (mg/l as CaCO <sub>3</sub> )     | 23                                | 58             |
| Total Residual Chlorine (mg/l)              | <q.l.< td=""><td>N/A</td></q.l.<> | N/A            |
| Ammonia (mg/l NH <sub>3</sub> -N)           | <1.0                              | N/A            |

Dilution water = Moderately hard synthetic freshwater made with ASTM Type I deionized water

| CBI Sample I.D. |              | Date(s)/Time(s)  1 <sup>st</sup> Used in Tests | Date(s)/Time(s) Used in Renewals | Samula Adia        |
|-----------------|--------------|--|----------------------------------|--------------------|
| ILUK0602-A      | 7/26/06 0930 | 7/26/06 1520, 1525                             | N/A                              | Sample Adjustments |

Client: Iluka Resources, Inc. Project ID: ILUK0602

Client Sample ID: Outfall 001 Permit No: VA0090981 Sample Period: 7/26/06



| Test:   | C. dubia 2002.0 |      |      |      |      | P    | . promelo | zs 2000.( | )    | SS-A attrocy |      |      |
|---------|-----------------|------|------|------|------|------|-----------|-----------|------|--------------|------|------|
| % Conc: | Cont.           | 6.3  | 13   | 25   | 50   | 100  | Cont.     | 6.3       | 13   | 25           | 50   | 100  |
| Temp.   | 25              | 25   | 25   | 25   | 25   | 25   | 25        | 25        | 25   | 25           | 25   | 25   |
| (°C)    | 0               | 0    | 0    | 0    | 0    | 0    | 0         | 0         | 0    | 0            | 0    | 0    |
| D.O.    | 8.1             | 8.1  | 8.1  | 8.0  | 8.0  | 8.0  | 8.0       | 8.0       | 8.0  | 7.9          | 7.9  | 7.8  |
| (mg/l)  | 0.1             | 0.1  | 0.2  | 0.2  | 0.2  | 0.2  | 0.2       | 0.2       | 0.2  | 0.2          | 0.3  | 0.4  |
| pH      | 7.76            | 7.73 | 7.73 | 7.73 | 7.67 | 7.64 | 7.54      | 7.60      | 7.60 | 7.61         | 7.56 | 7.58 |
| (S.U.)  | 0.07            | 0.07 | 0.06 | 0.05 | 0.05 | 0.33 | 0.13      | 0.06      | 0.07 | 0.08         | 0.14 | 0.39 |

| (Ref. Test Date)   | Data<br>Source | % Control<br>Survival | 48-h LC50 | rganism Source: CBL<br>95% C.L./A.L.<br>for LC50 | RTT in<br>Control? |
|--------------------|----------------|-----------------------|-----------|--|--------------------|
| C. dubia 2002.0    | RTT            | 100                   | 550       | 508-596  | Yes                |
| (7/17/06-7/19/06)  | CC             | 100                   | 569       | 514-624  | 103                |
| P. promelas 2000.0 | RTT            | 100                   | 910       | 868-955  | Yes                |
| (7/17/06-7/19/06)  | CC             | 99                    | 890       | 788-993  | 105                |

Note: RTT = Reference Toxicant Test, CC = Control Chart

The results of analysis contained within this report relate only to the sample as received in the laboratory. This report shall not be reproduced except in full without written approval from the laboratory.

APPROVED:

Peter F. De Lisle, Ph.D.

Technical Director

Ste/06 Date

#### GLOSSARY OF TERMS AND ABBREVIATIONS

A.L. (Acceptance Limits): The results of a given reference toxicant test are compared to the control chart mean value  $\pm 2$  standard deviations. These limits approximate the 95% probability limits for the "true" reference toxicant value.

C.L. (Confidence Limits): These are the probability limits, based on the data set and statistical model employed, that the "true value" lies within the limits specified. Typically limits are based on 95% or 99% probabilities.

Control chart: A cumulative summary chart of results from QC tests with reference toxicants. The results of a given reference toxicant test are compared to the control chart mean value and 95% Acceptance Limits (A.L.) (mean ± 2 standard deviations).

LC50: The concentration of sample or chemical, calculated from the data set using statistical models, causing a 50% reduction in test organism survival. The lower the LC50, the more toxic the chemical or sample. Units are same as test concentration units. Note: The LC50 value must always be associated with the duration of exposure. Thus 48-h LC50, 96-h LC50, etc. are calculated.

N/A: Not applicable. N/D: Not determined or measured.

NOAEC: No-observable-acute-effect-concentration. The highest concentration of sample or chemical in an acute test dilution series in which the test organisms exhibit no statistically significant reduction in the test end point (e.g. survival) compared to control organisms. Units are same as test concentration units.

Q.L.: Quantitation Limit. Level, concentration, or quantity of a target variable (analyte) that can be reported at a specified degree of confidence.

T.U.: Toxic units. Expresses the relative toxicity of an effluent in such a manner that the larger the toxic unit value the more toxic the effluent. T.U. $_{Ac} = 100/LC50$ . T.U. $_{Chr} = 100/NOEC$ . A dimensionless unit.

Page 2 of 2 Report Pages
Total No. Printouts/Bench Sheets Attached: 5

EPA Laboratory ID: VA01116

| NO | TES: |  |
|----|------|--|
|    |      |  |

| %<br>Effluent | I.D. | Day<br>0<br>Live | Day<br>1<br>Live | Day<br>2<br>Live | Final %<br>Survival | %<br>Effluent | I.D.   | Day<br>0<br>Live | Day<br>1<br>Live | Day<br>2<br>Live | Final %<br>Survival |
|---------------|------|------------------|------------------|------------------|---------------------|---------------|--------|------------------|------------------|------------------|---------------------|
|               | C-1  | 5                | 9                | 5                |                     |               | 3-1    | 5                | 5                | 5                |                     |
| Control       | C-2  | 5                | 5                | 5                |                     | ¥. /          | 3-2    | 5                | 5                | 4                |                     |
| 1             | C-3  | 5                | 5                | 5                | 100                 | 35            | 3-3    | 5                | 5                | 14               | 95                  |
|               | C-4  | 5                | 5                | 5                | ,                   |               | 3-4    | 5                | 5                | 14               |                     |
|               | 1-1  | 5                | 5                | 5                |                     |               | 4-1    | 5                | 5                | 8                |                     |
| 1. 3/         | 1-2  | 5                | 5                | 5                |                     | 500           | 4-2    | 5                | 5                | 3                |                     |
| 695           | 1-3  | 5                | 5                | 5                | 100                 | 50            | 4-3    | 5                | 5                | 3.               | 60                  |
|               | 1-4  | 5                | 5                | 5.               |                     |               | 4-4    | 5                | 5                | 4                |                     |
|               | 2-1  | 5                | 5                | 9                |                     | i             | 5-1    | 5                | 5                | 6                |                     |
| 12.5          | 2-2  | 5                | 5                | 5                | 100                 | 100           | 5-2    | 5                | 5                | )                |                     |
| (0.)          | 2-3  | 5                | 5                | 5                | , ,                 | 100           | 5-3    | 5                | 5                | 2                | 20                  |
|               | 2-4  | 5                | 5                | 5                |                     |               | 5-4    | 5                | 4                |                  |                     |
|               |      |                  |                  |                  |                     | Ini           | tials: | PB.              | CT               | LT               |                     |
|               |      |                  |                  |                  | L                   | Count T       | ime:   | 1525             | 0805             | 1919             | Test End<br>Time    |

|             | T                 | T              |             |       |
|-------------|-------------------|----------------|-------------|-------|
| Parameter   | Treatment<br>I.D. | Day 0          | Day 1       | Day 2 |
|             | С                 | 25             | 25          | 25    |
| Temp.       | 1                 | -5             | 25          | 25    |
| (°C)        | 2                 | 25             | 25          | 25    |
|             | 3                 | 25             | 25          | 85    |
|             | 4                 | 25             | 25          | 25    |
|             | 5                 | 25             | 75          | . 29  |
|             | С                 | 7.48           | 7.81        | 7.79  |
| pH          | 1                 | 7.46           | 7.74        | 7.79  |
| (S.U.)      | 2                 | 7.66           | 7.74        | 7.78  |
|             | 3.                | 7-69           | 7.32        | 7.78  |
|             | 4                 | 7.72           | 7638        | 7.67  |
|             | 5                 | 8.02           | 7.43        | 7.47  |
|             | С                 | 8.2            | 8.1         | 8.0   |
| D.O.        | İ                 | 4.2            | 8.1         | 8.0   |
| (mg/l)      | 2                 | 8,2            | 8.)         | 7.9   |
|             | 3                 | 8.2            | 8.0         | 7.8   |
|             | 4                 | 8.2            | 3.0         | 7.8   |
|             | 5                 | 8.2            | 8.0         | 7.8   |
|             | С                 | 291            | 4.7.7       | 292   |
| Conduct.    | 1                 | 705            |             |       |
| (uS/cm)     | 2                 | 1027           | 1979        |       |
|             | 3                 | 1422           |             | 5 7.5 |
| F           | 4                 | 1941           |             |       |
|             | .5                | 2670           | april 2004  | 2660  |
| Replicate # | Meas.:            | 1              | . 5         | 3     |
|             | nitials:          | 60             | CT          | . (7  |
| TRC (m      | g/l) in highe     | est conc. at e | nd of test: | , A   |
|             |                   |                |             | /1    |

| -         | managines Associated and Associated |
|-----------|---|
| Specie    | s: Ceriodaphnia dubia   |
| Source    | e: CBI stock cultures   |
|           | Other:  |
| Brood     |   |
| Releas    | e: Date/time end: 7/24/06 1016  |
| Acclim    | nation: Water: Mod. hard syn. FW  |
|           | Other   |
|           | Temperature (°C):25   |
| Feedin    | g: Prior to test: YCT/Selenastrum<br>During test: Not Fed   |
| Illumin   | nation: 16L:8D 10-20 uE/m²/s  |
| Test ch   | amber size:ml   |
| Solutio   | n volume:ml   |
| Numbe     | r of replicates/treatment:  |
| Initial r | number of daphnids/replicate:   |
| Randor    | nization template number: 24  |
| Set up:   | Date (Day 0): 7/26/06   |
|           | Time water added: 1506  |
|           | Time daphnids added: 1525   |
|           | Sat un by (initials): PB  |

| Stort Hoto:   |                        |        |         |                                | aapnnia | Test-48 Hr Surviva.                         |                                  |
|---|------------------------|--------|---------|--------------------------------|---------|---|----------------------------------|
| Start Date:<br>End Date:<br>Sample Date:<br>Comments: | 7/26/2006<br>7/28/2006 |        | Lab ID: | ILUK0602<br>CBI<br>EPAA 91-EPA | Acute   | Sample ID:<br>Sample Type:<br>Test Species: | 1<br>WW<br>CD-Ceriodaphnia dubia |
| Conc-%  | 1                      | 2      | 3       | 4                              |         | · · · · · · · · · · · · · · · · · · ·       |                                  |
| CONTROL   | 1.0000                 | 1.0000 | 1.0000  | 1.0000                         |         |   |                                  |
| 6.25  | 1.0000                 | 1.0000 | 1.0000  | 1.0000                         |         |   |                                  |
| 12.5  | 1.0000                 | 1.0000 | 1.0000  |                                |         |   |                                  |
| 25  | 1.0000                 | 0.8000 | 0.8000  | 0.8000                         |         |   |                                  |
| 50  | 0.4000                 | 0.6000 | 0.6000  | 0.8000                         |         |   |                                  |
| 100   | 0.0000                 | 0.2000 | 0.4000  | 0.2000                         |         |   |                                  |

| _                  |        | _      | Transform: Arcsin Square Root |        |        |        |        | Number  | Total |
|--------------------|--------|--------|-------------------------------|--------|--------|--------|--------|---------|-------|
| Conc-% Mean N-Mean | Mean   | Min    | Max                           | CV%    | N      |        | Number |         |       |
| CONTROL            | 1.0000 | 1.0000 | 1.3453                        | 1.3453 | 1,3453 | 0.000  | 4      | Resp Nu |       |
| 6.25               | 1.0000 | 1.0000 | 1.3453                        | 1.3453 | 1.3453 | 0.000  | 4      | 0       | 20    |
| 12.5               | 1.0000 | 1.0000 | 1.3453                        | 1.3453 | 1.3453 | 0.000  | 4      | 0       | 20    |
| 25                 | 0.8500 | 0.8500 | 1.1667                        | 1.1071 | 1.3453 | 10.206 | 4      | 0       | 20    |
| 50                 | 0.6000 | 0.6000 | 0.8910                        | 0.6847 | 1.1071 | 19.366 | 4      | 3       | 20    |
| 100                | 0.2000 | 0.2000 | 0.4594                        | 0.2255 | 0.6847 | 40.823 | 4      | 8       | 20    |
|                    |        |        | 001                           | 0.2200 | 0.0047 | 40.023 | 4      | . 16    | 20    |

| Auxillary Tests   | Statistic | Critical | Skew    | Kurt   |
|---|-----------|----------|---------|--------|
| Shapiro-Wilk's Test indicates non-normal distribution (p <= 0.01) | 0.75948   | 0.884    | 0.22775 |        |
| Equality of variance cannot be confirmed                          |           |          |         | 4,1010 |

| Equality of va | ariance can | not be cor | nfirmed                       |              |              |                        |         |         |            |         |      |
|----------------|-------------|------------|-------------------------------|--------------|--------------|------------------------|---------|---------|------------|---------|------|
| _              |             |            |                               | Maxi         | mum Likeliho | od-Probit              | 1       |         |            | -       |      |
| Parameter      | Value       | SE         | 95% Fldu                      | icial Limits | Control      |                        |         | P-value | Mu         | Sigma   | Iter |
| Slope          | 3.41157     | 0.66385    | 2.11042                       | 4.71272      | 0            | 0.6049                 | 7.81473 | 0.9     | 1.7544     | 0.29312 | 4    |
| Intercept      | -0.9852     | 1.13395    | -3.2078                       | 1.2373       |              |                        | 1101110 | 0.0     | 1.7044     | 0.28312 | 4    |
| TSCR           |             |            |                               |              |              | 1.0 7                  |         |         |            |         |      |
| Point          | Probits     | %          | 95% Fldu                      | icial Limits |              |                        |         |         | //         |         |      |
| EC01           | 2.674       | 11.8167    | The second name of the second | 18.3504      |              | 0.9                    |         |         | 1//        |         |      |
| EC05           | 3.355       | 18.7179    |                               | 26.1417      |              | 0.8                    |         |         | 11/        |         |      |
| EC10           | 3.718       | 23.9193    |                               | 31.8317      |              | 4                      |         |         | 17/        |         |      |
| EC15           | 3.964       | 28.2226    |                               | 36.5836      |              | 0.7 -                  |         |         | ///        | 1       |      |
| EC20           | 4.158       | 32.1885    |                               | 41.0945      |              | 0.6                    |         |         | ///        |         |      |
| EC25           | 4.326       |            | 25.6728                       |              |              | E                      |         | 1       | 1//        | 1       |      |
| EC40           | 4.747       |            | 37.0016                       |              |              | Response<br>0.5<br>0.4 |         | 1       | //         |         |      |
| EC50           | 5.000       |            | 44.786                        |              |              | Ø 0.4 −                |         | /4      | <b>\</b> / |         |      |
| EC60           | 5.253       | 67.3998    |                               |              |              | 1                      |         | //      | /          | 1       |      |
| EC75           | 5.674       | 89.5579    | 68.4189                       | 144.314      |              | 0.3                    |         | ///     |            |         |      |
| EC80           | 5.842       | 100.252    | 75.147                        | 171.092      |              | 0.2 -                  |         | ///     |            | 1       |      |
| EC85           | 6.036       | 114.34     | 83.5808                       | 209.258      |              | 0.1                    |         | /4/     |            |         |      |
| EC90           | 6.282       | 134.911    | 160 ISBN 기계 10 155            |              |              | 0.1                    |         | ///     |            |         |      |
| EC95           | 6.645       | 172.4      | 95.2405                       | 270.475      |              | 0.0 +                  |         | m       |            |         |      |
| EC99           | 7.326       |            |                               | 397.393      |              | 1                      |         | 10      | 100        | 1000    |      |
| _000           | 1.320       | 273.084    | 162.761                       | 824.441      |              |                        |         |         |            | .300    |      |
|                |             |            |                               |              |              |                        |         | Dose 9  | <b>%</b>   |         |      |

| %<br>Effluent | LD.     | Day 0<br>Live | Day 1<br>Live | Day 2<br>Live | Final %<br>Survival |
|---------------|---------|---------------|---------------|---------------|---------------------|
| Lab           | C-1     | 10            | to            | 10            |                     |
| Control       | C-2     | 10            | 10:           | 10            | 100                 |
| 625           | 1-1     | 10            | 10            | 10            |                     |
| 0,0)          | 1-2     | 10            | (0)           | 10            | 100                 |
| 11 ,-         | 2-1     | 10            | 10            | 10            |                     |
| 18.5          | 2-2     | 10            | 01            | (0            | 100                 |
| 2/            | 3-1     | 10            | 10            | 9             |                     |
| 25            | 3-2     | (0            | 10            | 10            | 95                  |
| 50            | 4-1     | 10            | 9             | 9             | 0.4                 |
| 3 —           | 4-2     | 10            | 9             | 9             | প্র                 |
| 100           | 5-1     | 10,           | 10            | 9             | 416                 |
| ( )           | 5-2     | (0            | 8             | 7             | 80                  |
|               | itials: | PB            | t.T           | CT            |                     |
| Count Time:   |         | 1500          | 0855          | 1510          | *Test End<br>Time   |

| Parameter   | Treatment I.D. | Day 0 | Day 1          | Day 2      |
|-------------|----------------|-------|----------------|------------|
|             | C              | 25    | 75             | 25         |
| Temp.       | 1              | 29    | 25             | 25         |
| (°C)        | 2              | 28    | 25             | 25         |
| l           | 3              | 25    | 25             | 25         |
|             | 4              | 25    | 25             | 25         |
|             | 5              | 25    | as             | 25         |
|             | C              | 7.68  | 7-42           | 7 53       |
| pН          | 1              | 7.46  | 7.55           | 7.61       |
| (S.U.)      | 2              | 246   | 7: 53          | 7.61       |
|             | 3              | 7-69  | 7.54           | 7.60       |
|             | 4              | 7.72  | 7.50           | 7.46       |
|             | 5              | 8.02  | 7.46           | 7. 23      |
|             | С              | 8.2   | 0,0            | 7.9        |
| D.O.        | 1              | 8-2   | 80             | 7,9        |
| (mg/l)      | 2              | 8.2   | 7.9            | 7.8        |
|             | 3              | 8.2   | 1.8            | 7.8        |
|             | 4              | 8-2   | 7.8            | 7.6        |
|             | 5              | 8.2   | 7.6            | 7.5        |
|             | С              | 291   | Market and the | 29)        |
| Conduct.    | 1              | 705   |                |            |
| (uS/cm)     | 2              | 1027  |                |            |
| [           | 3              | 1422  |                |            |
|             | 4              | 1961  |                | Address of |
|             | 5              | 2670  |                | 2736       |
| Replicate # | Meas.:         | 1     | a              | 1.         |
|             | Initials:      | A3    | (†             | ST         |
| TRC (n      | ng/l) in highe |       |                | MA         |
|             |                |       |                | 144        |

| COAST BIOANA | ALYSTS, INC          |     |
|--------------|----------------------|-----|
|              | EFFECTIVE DATE: 5/15 | 102 |

| Species: Pimephales promelas                                     |  |  |  |  |  |
|--|--|--|--|--|--|
| Source: CBI stock cultures                                       |  |  |  |  |  |
| Other:   |  |  |  |  |  |
|  |  |  |  |  |  |
| Hatch: Date/time start: 7/20/06 1100                             |  |  |  |  |  |
| Date /time end: 7/2/106 1100                                     |  |  |  |  |  |
| Acclimation: Water: Mod. hard syn. FW_                           |  |  |  |  |  |
| Other  |  |  |  |  |  |
| Temperature (°C): <u>A</u> S                                     |  |  |  |  |  |
| Feeding: Prior to test: Artemia ad libitum  During test: Not fed |  |  |  |  |  |
| Illumination: 16L:8D 10-20 uE/ $m^2$ /s                          |  |  |  |  |  |
| Test chamber size: 600 ml 250 ml ml                              |  |  |  |  |  |
| Solution volume: 500 ml ml                                       |  |  |  |  |  |
| Number of replicates/treatment: 2                                |  |  |  |  |  |
| Initial number of fish/replicate: / 💍                            |  |  |  |  |  |
| Randomization template number: /, 2                              |  |  |  |  |  |
| Set up: Date (Day 0): 7/26/06                                    |  |  |  |  |  |
| Time water added: ( 500  |  |  |  |  |  |
| Time fish added: 1520  |  |  |  |  |  |
| Set up by (initials):  |  |  |  |  |  |
| NOTES:   |  |  |  |  |  |

|                                      | INITIAI      | SAMPLE CHA  | RACTERIZATI  |                | A Principal Control of the Control o |             |
|--------------------------------------|--------------|-------------|--------------|----------------|--|-------------|
| Sample Bottle <sup>2</sup>           | AI           |             |              | NOTE           | S:   |             |
| Tot. Resid. Chlorine (mg/l)          | Chic         |             |              |                |  |             |
| Hardness (mg/l CaCO <sub>3</sub> )   | 1686         |             |              |                |  |             |
| Alkalinity (mg/l CaCO <sub>3</sub> ) | 23           |             |              |                |  |             |
| NH <sub>3</sub> -N (mg/l)            | . C1.D       |             |              |                |  |             |
| Color/Appearance <sup>3</sup>        | Ly           |             |              |                |  |             |
| Obvious Odor?                        | NO           |             |              |                |  |             |
| Date/Time                            | H261850      |             |              |                |  |             |
| Initials                             | an           |             |              |                |  |             |
|                                      | MPLE PREPARA | TION MEASUR | EMENTS (100° | concentration) |  |             |
| Sample Bottle                        | 0-1          |             | _            |                |  |             |
| Prep Temperature (°C)                | 25           |             |              |                |  |             |
| Conductivity (uS/cm)4                | 2670         |             |              | · .            |  |             |
| D.O. (mg/l) After Warming            | 8.2          |             |              |                |  |             |
| Aeration Time (min)                  | _            |             |              |                |  |             |
| Adjusted D.O.                        | -            |             |              |                |  |             |
| Final pH (S.U.)                      | 802          |             |              |                |  |             |
| Tot. Resid. Chlorine (mg/l)5         | N.O.         |             |              |                |  |             |
| Sample Filtered (60 um)?             | No           |             |              |                |  |             |
| Date/Time                            | 7/26/26/450  |             |              |                |  |             |
| Initials                             | PB           |             |              |                |  |             |
|                                      | DILUTI       | ON WATER CH | FARACTERIST  | (CS:           |  | <del></del> |
| Vat Number                           | 2.           |             |              |                |  |             |
| Temperature (°C)                     | 25           |             |              |                |  |             |
| Conductivity (uS/cm)                 | 291          |             |              |                |  |             |
| D.O. (mg/l)                          | 3.2          |             |              |                |  |             |
| pH (S.U.)                            | 768          |             |              |                |  |             |
| Hardness (mg/l CaCO <sub>3</sub> )   | 84           |             |              |                |  |             |
| Alkalinity (mg/l CaCO <sub>3</sub> ) | 58           |             |              |                |  |             |
| Date/Time                            | 7/26/91445   |             |              |                |  |             |
| Initials                             | PB           |             |              |                |  |             |

(First 8 characters of Laboratory Sample ID)

<sup>&</sup>lt;sup>1</sup>Q.L. = Quantification Limit, N.D. = Not Determined/Measured, NA = Not Applicable

<sup>2</sup>Bottle number = 9<sup>th</sup> or 9<sup>th</sup> and 10<sup>th</sup> characters of Laboratory Sample I.D. (e.g. "A", "A-1", "A-2"; see SOP SPLS202)

<sup>3</sup>C-Clear, O-Opaque, T-Turbid, S-Solids (SI-Slight, M-Moderate, H-Heavy), Y-Yellow, B-Brown, B1-Black, G-Green

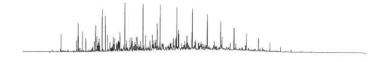
<sup>&</sup>lt;sup>4</sup>Conductivity measured on first use of sample only

<sup>&</sup>lt;sup>5</sup>Total residual chlorine measured after sample prep only if present in initial sample characterization

ITEM 2

## Primary Laboratories, Inc.

7423 Lee Davis Road • Mechanicsville, VA 23111 • Telephone (804) 559-9004 • Fax (804) 559-9306



## ANALYTICAL LABORATORY REPORT

13-Dec-06

Iluka Resources, Inc Attn: Kevin Rideout 12472 St Johns Church Road Stoney Creek, VA 23882

Date Received:

21-Nov-06

Date Sampled:

21-Nov-06

Work Order No:

0611185-01

Client ID:

#1 - #11

| Client ID:   | #1 - #11  |  |  |   |   |   |                                  |
|--|---|--|--|---|---|---|----------------------------------|
| Test   | Final   | Reporting  | Units of   | EPA Test  | Date  | Time  | Tech.                            |
| Description  | Result  | Limit  | Measure  | Method  | Analyzed  | Analyzed  | Initials                         |
| Dissolved Metals Antimony Arsenic Cadmium Mercury Nickel Selenium Silver | <0.100<br><0.050<br><0.010<br><0.0002<br>0.021<br>0.064<br><0.020 | 0.100<br>0.050<br>0.010<br>0.0002<br>0.020<br>0.050<br>0.020 | mg/L<br>mg/L<br>mg/L<br>mg/L<br>mg/L<br>mg/L<br>mg/L | 200.2/200.7<br>200.2/200.7<br>200.2/200.7<br>245.1<br>200.2/200.7<br>200.2/200.7<br>200.2/200.7 | 04-Dec-06<br>29-Nov-06<br>29-Nov-06<br>30-Nov-06<br>29-Nov-06<br>04-Dec-06<br>29-Nov-06 | 15:11<br>15:59<br>15:59<br>15:37<br>15:59<br>15:11<br>15:59 | AB<br>AB<br>AB<br>AB<br>AB<br>AB |
| Zinc   | 0.044   | 0.010  | mg/L   | 200.2/200.7   | 29-Nov-06   | 15:59   | AB                               |
| Chromium   | <0.020  | 0.020  | mg/L   | 200.2/200.7   | 29-Nov-06   | 15:59   | AB                               |
| Chromium III<br>Chromium VI  | <0.020<br><0.005  | 0.020<br>0.005   | mg/L<br>mg/L   | calc.<br>218.4  | 13-Dec-06<br>22-Nov-06  | 11:00   | PB<br>NA                         |
| Cyanide  | <0.010  | 0.010  | mg/L   | 335.2   | 28-Nov-06   | 11:00   | MS                               |
| Hydrogen Sulfide   | <0.05   | 0.05   | mg/L   | 376.1   | 04-Dec-06   | 15:20   | HV                               |
| Fecal Coliform   | 2   | 2  | MPN/100ml  | 9221E   | 21-Nov-06   | 17:00   | MS                               |
| Chlorides  | 14.1  | 0.1  | mg/L   | 325.3   | 30-Nov-06   | 15:30   | NA                               |
| Chlorine, Total Residual   | <0.010  | 0.010  | mg/L   | 330.5   | 22-Nov-06   | 14:50   | HV                               |
| Tributyltin  | <30   | 30   | ng/L   | GC/FID  | 01-Dec-06   | 16:09   | SC*                              |

## Primary Laboratories, Inc.

Results

13-Dec-06

Date Sampled: Work Order No:

21-Nov-06 0611185-01

Client ID:

#1 - #11

| Client ID:         | #1 - #11 |           |          |          |           |          |          |
|--------------------|----------|-----------|----------|----------|-----------|----------|----------|
| Test               | Final    | Reporting | Units of | EPA Test | Date      | Time     | Tech.    |
| Description        | Result   | Limit     | Measure  | Method   | Analyzed  | Analyzed | Initials |
| 15                 |          |           |          |          |           |          |          |
| Pesticides         |          |           |          |          |           |          |          |
| Aldrin             | <0.05    | 0.05      | ug/L     | 608      | 22-Nov-06 | 22:24    | HV       |
| Chlordane          | <0.20    | 0.20      | ug/L     | 608      | 22-Nov-06 | 22:24    | HV       |
| Dieldrin           | <0.10    | 0.10      | ug/L     | 608      | 22-Nov-06 | 22:24    | HV       |
| 4,4-DDT            | < 0.10   | 0.10      | ug/L     | 608      | 22-Nov-06 | 22:24    | HV       |
| 4,4-DDE            | <0.10    | 0.10      | ug/L     | 608      | 22-Nov-06 | 22:24    | HV       |
| 4,4-DDD            | <0.10    | 0.10      | ug/L     | 608      | 22-Nov-06 | 22:24    | HV       |
| Endosulfan sulfate | <0.10    | 0.10      | ug/L     | 608      | 22-Nov-06 | 22:24    | HV       |
| Endosulfan I       | < 0.10   | 0.10      | ug/L     | 608      | 22-Nov-06 | 22:24    | HV       |
| Endosulfan II      | < 0.10   | 0.10      | ug/L     | 608      | 22-Nov-06 | 22:24    | HV       |
| Endrin             | < 0.10   | 0.10      | ug/L     | 608      | 22-Nov-06 | 22:24    | HV       |
| Gamma-BHC (Lindane | < 0.50   | 0.50      | ug/L     | 608      | 22-Nov-06 | 22:24    | HV       |
| Beta-BHC           | < 0.50   | 0.50      | ug/L     | 608      | 22-Nov-06 | 22:24    | HV       |
| Alpha-BHC          | < 0.50   | 0.50      | ug/L     | 608      | 22-Nov-06 | 22:24    | HV       |
| Heptachlor         | < 0.50   | 0.50      | ug/L     | 608      | 22-Nov-06 | 22:24    | HV       |
| Kepone             | <0.10    | 0.10      | ug/L     | 608      | 22-Nov-06 | 22:24    | HV       |
| Methoxychlor       | <0.10    | 0.10      | ug/L     | 608      | 22-Nov-06 | 22:24    | HV       |
| Mirex              | <0.10    | 0.10      | ug/L     | 608      | 22-Nov-06 | 22:24    | HV       |
| Endrin Aldehyde    | < 0.10   | 0.10      | ug/L     | 608      | 22-Nov-06 | 22:24    | HV       |
| Heptachlor Epoxide | <0.10    | 0.10      | ug/L     | 608      | 22-Nov-06 | 22:24    | HV       |
| PCB 1016           | <1.0     | 1.0       | ug/L     | 608      | 22-Nov-06 | 22:24    | HV       |
| PCB 1221           | <1.0     | 1.0       | ug/L     | 608      | 22-Nov-06 | 22:24    | HV       |
| PCB 1232           | <1.0     | 1.0       | ug/L     | 608      | 22-Nov-06 | 22:24    | HV       |
| PCB 1242           | <1.0     | 1.0       | ug/L     | 608      | 22-Nov-06 | 22:24    | HV       |
| PCB 1248           | <1.0     | 1.0       | ug/L     | 608      | 22-Nov-06 | 22:24    | HV       |
| PCB 1254           | <1.0     | 1.0       | ug/L     | 608      | 22-Nov-06 | 22:24    | HV       |
| PCB 1260           | <1.0     | 1.0       | ug/L     | 608      | 22-Nov-06 |          | HV       |
| Toxaphene          | <5.0     | 5.0       | ug/L     | 608      | 22-Nov-06 | 22:24    | HV       |

Date Sampled: Work Order No:

21-Nov-06 0611185-01

#1 - #11

| Client ID:                     | #1 - #11 |           |          |          |           |          |          |
|--------------------------------|----------|-----------|----------|----------|-----------|----------|----------|
| Test                           | Final    | Reporting | Units of | EPA Test | Date      | Time     | Tech.    |
| Description                    | Result   | Limit     | Measure  | Method   | Analyzed  | Analyzed | Initials |
| Organophosphorus<br>Pesticides |          |           |          |          |           |          |          |
| Demeton                        | <1       | 1         | ug/L     | 622      | 12-Dec-06 | 19:01    | SC*      |
| Malathion                      | <1       | 1         | ug/L     | 622      | 12-Dec-06 | 19:01    | SC*      |
| Chlorpyrifos                   | <0.2     | 0.2       | ug/L     | 622      | 12-Dec-06 | 19:01    | SC*      |
| Parathion                      | <1       | 1         | ug/L     | 622      | 12-Dec-06 | 19:01    | SC*      |
| Guthion                        | <1       | 1         | ug/L     | 622      | 12-Dec-06 | 19:01    | SC*      |
|                                | 1        |           |          |          |           |          |          |

# Primary Laboratories, Inc. Results

13-Dec-06

| Test Method:      | 624        |
|-------------------|------------|
| Date Analyzed:    | 28-Nov-06  |
| Time Analyzed:    | 18:10      |
| Technician:       | PB         |
| Date Sampled:     | 21-Nov-06  |
| Units of Measure: | ug/L       |
| Work Order No:    | 0611185-01 |
| Client ID:        | #1 - #11   |

| WOIN CIGOT ING.       | 00111000 |           |
|-----------------------|----------|-----------|
| Client ID:            | #1 - #11 |           |
| Test                  | Final    | Detection |
| Description           | Result   | Limit     |
|                       |          |           |
| Acrolein              | <5       | 5         |
| Acrylonitrile         | <5       | 5         |
| Benzene               | <5       | 5         |
| Bromoform             | <5       | 5         |
| Carbon tetrachloride  | <5       | 5         |
| Chlorobenzene         | <5       | 5         |
| Chlorodibromometha    | nne <5   | 5         |
| Chloroform            | <5       | 5         |
| Dichloromethane       | <5       | 5         |
| Dichlorobromometha    | ine <5   | 5         |
| 1,1-Dichloroethylene  | <5       | 5         |
| 1,2-Dichloroethane    | <5       | 5         |
| 1,2-trans-Dichloroeth |          | 5         |
| 1,2-Dichloropropane   | <5       | 5         |
| 1,3-Dichloropropene   | <5       | 5         |
| Ethylbenzene          | <5       | 5         |
| Methylene Bromide     | <5       | 5         |
| 1,1,2,2-Tetrachloroet | thane <5 | 5         |
| Tetrachloroethylene   | <5       | 5         |
| Toluene               | <5       | 5         |
| Trichloroethylene     | <5       | 5         |
| 1,1,2-Trichloroethane | e <5     | 5         |
| Vinyl Chloride        | <5       | 5         |
| Monochlorobenzene     | <5       | 5         |
| Xylenes (Total)       | <15      | 15        |
|                       |          |           |
|                       |          |           |

# Primary Laboratories, Inc. Results

13-Dec-06

| Test Method:      | 625        |
|-------------------|------------|
| Date Analyzed:    | 22-Nov-06  |
| Time Analyzed:    | 22:02      |
| Technician:       | HV         |
| Date Sampled:     | 21-Nov-06  |
| Units of Measure: | ug/L       |
| Work Order No:    | 0611185-01 |
| Client ID:        | #1 #11     |

|    | Client ID:                | #1-#11 |           |
|----|---------------------------|--------|-----------|
|    | Test                      | Final  | Detection |
|    | Description               | Result | Limit     |
|    |                           |        |           |
| J  | Acenaphthene              | <10    | 10        |
|    | Anthracene                | <10    | 10        |
| ?  | Benzo(a) anthracene       | <10    | 10        |
| è  | Benzo(b) fluoranthene     | <10    | 10        |
| 7  | Benzo(k) fluoranthene     | <10    | 10        |
|    | Benzo(a)pyrene            | <10    | 10        |
|    | Butyl benzyl phthalate    | <10    | 10        |
| L  | 2-Chlorophenol            | <10    | 10        |
| -  | Chrysene                  | <10    | 10        |
|    | Dibenzo(a,h)anthracene    | <10    | 20        |
|    | Di-n-butyl phthalate      | <10    | 10        |
|    | 1,2-Dichlorobenzene       | <10    | 10        |
|    | 1,3-Dichlorobenzene       | <10    | 10        |
|    | 1,4-Dichlorobenzene       | <10    | 10        |
| V. | 2,4-Dichlorophenol        | <10    | 10        |
|    | Diethyl phthalate         | <10    | 10        |
|    | 2,4-Dimethylphenol        | <10    | 10        |
|    | Di-2-Ethylhexyl Phthalate | <10    | 10        |
| /  | 2,4-Dinitrotoluene        | <10    | 10        |
|    |                           |        |           |

# Primary Laboratories, Inc. Results

13-Dec-06

| Test Method:      | 625 (Con't) |
|-------------------|-------------|
| Date Analyzed:    | 22-Nov-06   |
| Time Analyzed:    | 22:02       |
| Technician:       | HV          |
| Date Sampled:     | 21-Nov-06   |
| Units of Measure: | ug/L        |
| Work Order No:    | 0611185-01  |
| Client ID:        | #1 - #11    |

| Client ID:              | #1 - #11 |           |
|-------------------------|----------|-----------|
| Test                    | Final    | Detection |
| Description             | Result   | Limit     |
| Fluoranthene            | <10      | 10        |
| Fluorene                | <10      | 10        |
| Indeno(1,2,3-cd) pyrene | <20      | 20        |
| Isophorone              | <10      | 10        |
| Nitrobenzene            | <10      | 10        |
| Pentachlorophenol       | <10      | 10        |
| Phenol                  | <10      | 10        |
| Pyrene                  | <10      | 10        |
| 1,2,4-Trichlorobenzene  | <10      | 10        |
| 2,4,6-Trichlorophenol   | <10      | 10        |
| Naphthalene             | <10      | 10        |
| 29                      |          |           |

Signature: 🛕

Parry L. Bragg

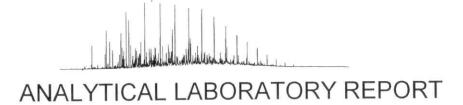
Laboratory Manager /

nded for the exclusive use of the client. These

These analytical results are based upon materials provided by the client and are intended for the exclusive use of the client. These analytical results represent the best judgement of Primary Laboratories, Inc. Primary Laboratories, Inc. assumes no responsibility, express or implied, as to the interpretation of the analytical results contained in this report. This report is not to be reproduced except with the written approval of Primary Laboratories, Inc.

# Primary Laboratories, Inc.

7423 Lee Davis Road • Mechanicsville, VA 23111 • Telephone (804) 559-9004 • Fax (804) 559-9306



15-May-07

Iluka Resources, Inc Attn: Kevin Rideout

12472 St Johns Church Road Stoney Creek, VA 23882

Date Received:

2-Apr-07

Date Sampled:

2-Apr-07

Work Order No:

0704009-01

| Client ID:   | Outfall 001 (M     | ISP) Renewa    |          |             |  |              |          |
|--|--------------------|----------------|----------|-------------|--|--------------|----------|
| Test   | Final              | Reporting      | Units of | EPA Test    | Date   | Time         | Tech.    |
| Description  | Result             | Limit          | Measure  | Method      | Analyzed   | Analyzed     | Initials |
|  |                    |                |          |             |  |              |          |
| Dissolved Metals   |                    | INC. APPRINCES |          |             |  | 15.01        |          |
| Copper   | <0.020             | 0.020          | mg/L     | 200.2/200.7 | 5-Apr-07   | 15:21        | AB       |
| Lead   | <0.050             | 0.050          | mg/L     | 200.2/200.7 |  | 15:21        | AB       |
| Thallium   | <0.002             | 0.002          | mg/L     | 200.2/279.2 | Control of the contro | 11:21        | AB       |
| Nickel   | <0.020             | 0.020          | mg/L     | 200.2/200.7 | 5-Apr-07   | 15:21        | AB       |
| Selenium   | < 0.050            | 0.050          | mg/L     | 200.2/200.7 | 5-Apr-07   | 15:21        | AB       |
| Zinc   | 0.041              | 0.010          | mg/L     | 200.2/200.7 | 5-Apr-07   | 15:21        | AB       |
|  |                    |                |          |             |  |              |          |
| BOD  | <3.0               | 3.0            | mg/L     | 405.1       | 4-Apr-07   | 07:30        | NA       |
| *  |                    |                |          |             |  |              |          |
| COD  | 22.1               | 2.0            | mg/L     | 410.2       | 16-Apr-07  | 08:30        | NA       |
|  | -                  |                |          |             |  |              |          |
| тос  | 5.38               | 1.00           | mg/L     | 415.1       | 3-Apr-07   | 07:30        | PB       |
|  |                    |                |          |             |  |              |          |
| Gross Alpha  | 6.0 <u>+</u> 0.7   | 0.4            | pci/l    | 900.0       | 20-Apr-07  | 08:00        | SC*      |
|  |                    |                |          |             |  |              |          |
| Gross Beta   | 21 <u>+</u> 3      | 5.7            | pci/l    | 900.0       | 19-Apr-07  | 08:00        | SC*      |
| The second secon | No.                |                |          |             |  |              |          |
| Strontium-90   | 0.0 <u>+</u> 0.5   | 1.0            | pci/l    | 905.0       | 23-Apr-07  | 9:23         | SC*      |
|  |                    |                |          |             |  | 1000 percent |          |
| Tritium  | 30.1 <u>+</u> 87.2 | 144.1          | pci/l    | 906.0       | 1-May-07   | 12:00        | SC*      |

<sup>\*</sup> Sub-Contracted

# Primary Laboratories, Inc. Results

15-May-07

Date Sampled:

2-Apr-07

Work Order No:

0704009-01

Client ID:

Outfall 001 (MSP) Renewal

| Client ID. Outlan 601 (MSF) Kenewai |             |           |          |          |           |          |          |
|-------------------------------------|-------------|-----------|----------|----------|-----------|----------|----------|
| Test                                | Final       | Reporting | Units of | EPA Test | Date      | Time     | Tech.    |
| Description                         | Result      | Limit     | Measure  | Method   | Analyzed  | Analyzed | Initials |
|                                     |             |           |          |          |           |          |          |
| Pesticides                          |             |           |          |          |           |          |          |
| Gamma-BHC (Lindane                  | <0.50       | 0.50      | ug/L     | 608      | 10-Apr-07 | 23:57    | HV       |
| Methoxychlor                        | <0.10       | 0.10      | ug/L     | 608      | 10-Apr-07 | 23:57    | HV       |
| PCB 1016                            | <1.0        | 1.0       | ug/L     | 608      | 10-Apr-07 | 23:57    | HV       |
| PCB 1221                            | <1.0        | 1.0       | ug/L     | 608      | 10-Apr-07 | 23:57    | HV       |
| PCB 1232                            | <1.0        | 1.0       | ug/L     | 608      | 10-Apr-07 | 23:57    | HV       |
| PCB 1242                            | <1.0        | 1.0       | ug/L     | 608      | 10-Apr-07 | 23:57    | HV       |
| PCB 1248                            | <1.0        | 1.0       | ug/L     | 608      | 10-Apr-07 | 23:57    | HV       |
| PCB 1254                            | <1.0        | 1.0       | ug/L     | 608      | 10-Apr-07 | 23:57    | HV       |
| PCB 1260                            | <1.0        | 1.0       | ug/L     | 608      | 10-Apr-07 | 23:57    | HV       |
|                                     | 100,110,000 |           |          |          |           |          |          |
|                                     |             |           | -        |          |           |          |          |

# Primary Laboratories, Inc. Results

15-May-07

| Test Method:      | 625        |
|-------------------|------------|
| Date Analyzed:    | 4-Apr-07   |
| Time Analyzed:    | 16:35      |
| Technician:       | HV         |
| Date Sampled:     | 2-Apr-07   |
| Units of Measure: | ug/L       |
| Work Order No:    | 0704009-01 |

| Client ID:   | Outfall 001 | (MSP) | Renewal |
|--|-------------|-------|---------|
| Control of the Contro |             | 1     |         |

| Client ID:                    | Outfall 001 (N | ISP) Renewa |
|-------------------------------|----------------|-------------|
| Test                          | Final          | Detection   |
| Description                   | Result         | Limit       |
|                               |                |             |
| Benzidine                     | <10            | 10          |
| bis-(2-Chloroethyl)ether      | <10            | 10          |
| bis-(2-Chloroisopropyl )ether | <10            | 10          |
| 2-Chloronaphthalene           | <10            | 10          |
| 3,3-Dichlorobenzidine         | <20            | 20          |
| Dimethyl phthalate            | <10            | 10          |
| 2,4-Dinitrophenol             | <10            | 10          |
| Hexachlorobenzene             | <10            | 10          |
| Hexachlorobutadiene           | <10            | 10          |
| Hexachlorocyclopentadiene     | <10            | 10          |
| Hexachloroethane              | <10            | 10          |
| Indeno(1,2,3-cd) pyrene       | <20            | 20          |
| 2-Methyl-4,6-Dinitrophenol    | <50            | 50          |
| N-Nitrosodimethylamine        | <10            | 10          |
| N-Nitrosodiphenylamine        | <10            | 10          |
| N-Nitrosodi-n-propylamine     | <10            | 10          |
| 1,2-DiphenIhrazine            | <10            | 10          |
|                               |                |             |

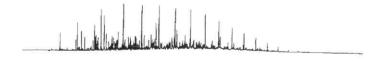
Signature: Date: 5/15/07

Parry L. Bragg
Laboratory Manager

These analytical results are based upon materials provided by the client and are intended for the exclusive use of the client. These analytical results represent the best judgement of Primary Laboratories, Inc. Primary Laboratories, Inc. assumes no responsibility, express or implied, as to the interpretation of the analytical results contained in this report. This report is not to be reproduced except with the written approval of Primary Laboratories, Inc.

# Primary Laboratories, Inc.

7423 Lee Davis Road • Mechanicsville, VA 23111 • Telephone (804) 559-9004 • Fax (804) 559-9306



# ANALYTICAL LABORATORY REPORT

26-Jun-07

Iluka Resources, Inc Attn: Kevin Rideout

12472 St Johns Church Road Stoney Creek, VA 23882

Project:

MSP Permit Renewal

Date Received:

19-Jun-07

Date Sampled:

18-Jun-07

Work Order No:

0706164-01

Client ID:

Outfall 001

| Outtail 001     |                                 |   |  |   |  |  |
|-----------------|---------------------------------|---|--|---|--|--|
| Final<br>Result | Reporting<br>Limit              | Units of<br>Measure                               | EPA Test<br>Method   | Date<br>Analyzed                        | Time<br>Analyzed   | Tech.  |
| 0.02            | 0.01                            | mg/L  | 350.3  | 25-Jun-07                               | 08:00  | NA   |
| 19.5            | 0.1                             | mg/L  | 325.3  | 26-Jun-07                               | 09:00  | NA   |
| <0.05           | 0.05                            | mg/L  | 376.1  | 19-Jun-07                               | 13:30  | NA   |
|                 | Final<br>Result<br>0.02<br>19.5 | Final Reporting Result Limit  0.02 0.01  19.5 0.1 | Final Reporting Units of Result Limit Measure  0.02 0.01 mg/L  19.5 0.1 mg/L | Final   Reporting   Units of   EPA Test | Final Reporting Result         Reporting Limit         Units of Measure         EPA Test Method         Date Analyzed           0.02         0.01         mg/L         350.3         25-Jun-07           19.5         0.1         mg/L         325.3         26-Jun-07 | Final Reporting Result         Reporting Limit         Units of Measure         EPA Test Method         Date Analyzed         Time Analyzed           0.02         0.01         mg/L         350.3         25-Jun-07         08:00           19.5         0.1         mg/L         325.3         26-Jun-07         09:00 |

Laboratory Manager

These analytical results are based upon materials provided by the client and are intended for the exclusive use of the client. These analytical results represent the best judgement of Primary Laboratories, Inc. Primary Laboratories, Inc. assumes no responsibility, express or implied, as to the interpretation of the analytical results contained in this report. This report is not to be reproduced except with the written approval of Primary Laboratories, Inc.

ITEM 3



2960 Foster Creighton Dr ashville, TN 37204 o15-726-0177 Fax: 615-726-0954

#### ANALYTICAL REPORT

ILUKA RESOURCES INC. 9874 CHEE SAUNDERS 12472 ST. JOHN CHURCH RD. STONY CREEK, VA 23882-0129

Project:

Project Name: VPDES Sampler: CHEE SAUNDERS Lab Number: 00-A114305

Sample ID: ZIRCON

Sample Type: Solid waste

Site ID:

Date Collected: 8/14/00 Time Collected: 13:45 Date Received: 8/15/00 Time Received: 10:00

#### TCLP Results

|   |           |        | 81        | atrix Spike                                 |         |       |             |                |   |
|---|-----------|--------|-----------|---|---------|-------|-------------|----------------|---|
| Analyte   | Result    | Units  | Reg Linit | Recovery (%)                                | Date    | Tine  | Analyst     | Method         | QC Batch                                |
| party and that then then seen and seel have now man man and also also are due to the hour has been man that were<br>The |           |        |           | but the net bee one par bee one per had yet |         |       |             |                | *************************************** |
| Arsenic   | ( 0.100   | Hq/1   | 5.0       | 186   | 8/22/00 | 7:52  | G. Robinson | (0100          | 9937                                    |
|   | ( 1.00    | tige 1 | 100       | 113   | 8/22/80 | 7:52  | 6. Robinson | 60108<br>60108 | 8236<br>8236                            |
| ATTURE TOTAL  | ( 0.100   | Hg/1   | 1.0       | 100   | 8/22/00 | 7:52  | G. Robinson | 60108          | 8236                                    |
| aronium   | 0.500     | Hg/1   | 5.8       | 103   | 8/22/00 | 7:52  | G. Robinson | 60108          | 8236                                    |
| Lead  | 0.500     | ng/1   | 5.0       | 98  | 8/22/00 | 7:52  | 6. Robinson | 60108          | 8236                                    |
| Mercury   | 0.0100    | tig/L  | 0.20      | 100   | 6/22/00 | 7:58  | 6. NoCord   | 74788          | 9068                                    |
| Selenium  | 0.100     | Hg/1   | 1.0       | 115   | 8/22/00 | 7:52  | 6. Robinson | 60108          | 8236                                    |
| Silver  | ( 0.100   | ng/1   | 5.0       | 105   | 8/22/00 | 7:52  | G. Robinson | 60108          | 8236                                    |
| TCLP Extraction   | Initiated |        |           |   | 8/16/00 | 17:30 | K. Bundy    | 1311           | 6948                                    |

ND = Not detected at the report limit.

Matrix Spike Recovery for TCLP analytes reported from Datch QC
TCLP prepartion follows method 1311, SN-846 Revision 3.

All results reported on a met meight basis.



2960 Foster Creighton Dr ashville, TN 37204 o15-726-0177 Fax: 615-726-0954

#### ANALYTICAL REPORT

Laboratory Number: 00-A114305

Sample ID: ZIRCON

Page 2

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Report Approved By:

Paul E. Lane, Jr., Lab Director Michael H. Dunn, M.S., Technical Director Johnny A. Mitchell, Dir. Technical Serv. Eric S. Smith, Assistant Technical Director Report Date: 8/22/00

Gail A. Lage, Technical Serv. Glenn L. Morton, Technical Serv. Kelly S. Comstock, Technical Serv. Pamela A. Langford, Technical Serv.

Laboratory Certification Number: 00323



2960 Foster Creighton Dr Nashville, TN 37204 515-726-0177 Fax: 615-726-0954

#### ANALYTICAL REPORT

ILUKA RESOURCES INC. 9874 CHEE SAUNDERS 12472 ST. JOHN CHURCH RD. STONY CREEK, VA 23882-0129

Project:

Project Name: VPDES Sampler: CHEE SAUNDERS Lab Number: 00-A114304 Sample ID: ILMENITE Sample Type: Solid waste

Site ID:

site in.

Date Collected: 8/14/00 Time Collected: 13:45 Date Received: 8/13/00 Time Received: 10:00

#### TCLP Results

| Matrix Spike    |  |                                |           |   |  |        |             |        |          |
|-----------------|--|--------------------------------|-----------|---|--|--------|-------------|--------|----------|
| Analyte         | Result                                       | Units                          | Reg Linit | Recovery (%)                                | Date   | Tine   | Analyst     | Method | AC Ratch |
| *               | and was not and and not now are aim ago pio- | THE PERSON NAME AND ADDRESS OF |           | 249 506 500 500 500 500 600 500 500 500 500 | . 10-1 20-1 100 20-1 100 510 510 100 100 100 | -      |             |        |          |
| Arsenio         | 0.100  | ng/1                           | 5.0       | 106   | 8/22/00                                      | 7:52   | 6. Robinson | 6010K  | 8236     |
| Harium          | ( 1.00                                       | ng/1                           | 100       | 113   | 8/22/00                                      | 7:52   | 6. Robinson | 6010B  | 8236     |
| Cadniun         | 0.100  | mg/1                           | 1.0       | 100   | 8/22/00                                      | 7:52   | 6. Robinson | 6010B  | 8236     |
| Chroniun        | ( 0.500                                      | Hg/1                           | 5.0       | 103   | 8/22/00                                      | 7:52   | 6. Robinson | 60100  | 8236     |
| Lead            | 0.500  | ng/1                           | 5.0       | 98  | 8/22/00                                      | 7:52   | 6. Robinson | 6010B  | 8236     |
| Hercury         | 0.0100                                       | ng/1                           | 0.20      | 100   | 8/22/00                                      | 7:58   | 6. NoCord   | 7470A  | 9068     |
| Selenium        | 0.100  | ng/1                           | 1.0       | 115   | 8/22/00                                      | 7:52   | 6. Robinson | 6010B  | 8236     |
| Silver          | 0.100  | mg/1                           | 5.0       | 105   | 8/22/00                                      | 7:52   | 6. Robinson | 6010B  | 8236     |
| TCLF Extraction | Initiated                                    |                                |           |   | 8/16/00                                      | 17: 30 | K. Rundy    | 1311   | 6948     |

HD = Not detected at the report limit.

Matrix Spike Recovery for TCLP analytes reported from Batch QC
TCLP prepartion follows method 1311, SW-846 Revision 3.

All results reported on a met meight basis.



2960 Foster Creighton Dr Nashville, TN 37204 615-726-0177 Fax: 615-726-0954

#### ANALYTICAL REPORT

Laboratory Number: 00-A114304

Sample ID: ILMENITE

Page 2

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Report Approved By

Report Date: 8/22/00

Faul E. Lane, Jr., Lab Director Michael H. Dunn, M.S., Technical Director Johnny A. Mitchell, Dir. Technical Serv. Eric S. Smith, Assistant Technical Director

Gail A. Lage, Technical Serv. Glenn L. Norton, Technical Serv. Kelly S. Comstock, Technical Serv. Famela A. Langford, Technical Serv.

Laboratory Certification Number: 00323



2960 Foster Creighton Dr Nashville, TN 37204 615-726-0177 Fax: 615-726-0954

#### ANALYTICAL REPORT

ILUKA RESOURCES INC. 9874 CHEE SAUNDERS 12472 ST. JOHN CHURCH RD. STONY CREEK, VA 23882-0129

Project:

Project Name: VPDES Sampler: CHEE SAUNDERS Lab Number: 00-A114303

Sample ID: MINERAL SAND CONCENTRATE

Sample Type: Solid waste

Site ID:

Date Collected: 8/14/00 Time Collected: 13:45 Date Received: 8/15/00 Time Received: 10:00

#### TCLP Results

|   |   |   |       | 1         | atrix Spike  |         |       |  |        |          |
|---|---|---|-------|-----------|--------------|---------|-------|--|--------|----------|
|   | Analyte                                 | Result  | Units | Reg Linit | Recovery (Z) | Date    | Tine  | Analyst  | Method | QC Batch |
| 4 | AND | and \$400 for man and paperary and add you have |       |           |              |         |       | MRS 1000 THE MRS NAME WAS STREET, AND ADDRESS. |        | ****     |
|   | Arsenic                                 | ₹ 0.100   | ng/1  | 5.0       | 106          | 8/22/00 | 7:52  | 6. Robinson                                    | 60108  | 8236     |
|   | Barium                                  | ₹ 1.00  | ng/1  | 100       | 113          | 8/22/00 | 7:52  | G. Robinson                                    | 6010B  | 8236     |
|   | Cadniun                                 | < 0.100   | ng/1  | 1.0       | 100          | 8/22/00 | 7:52  | G. Robinson                                    | 60108  | 8236     |
|   | Chronium                                | ₹ 0.500   | tig/1 | 5.0       | 103          | 8/22/00 | 7:52  | G. Robinson                                    | 60108  | 8236     |
|   | Lead                                    | ₹ 0.500   | ng/1  | 5.0       | 98           | 8/22/00 | 7:52  | G. Robinson                                    | 60108  | 8236     |
|   | Mercury                                 | < 0.0100  | ng/1  | 0.20      | 100          | 8/22/00 | 7:58  | S. NcCord                                      | 7470A  | 9068     |
|   | Selenium                                | < 0.100   | mg/1  | 1.0       | 115          | 8/22/00 | 7:52  | G. Robinson                                    | 60108  | 8236     |
|   | Silver                                  | ₹ 0.100   | Hq/1  | 5.0       | 105          | 8/22/00 | 7:52  | G. Robinson                                    | 6010B  | 8236     |
|   | TCLP Extraction                         | Initiated                                       | -     |           |              | 8/16/00 | 17:30 | K. Bundy                                       | 1311   | 6948     |
|   |   |   |       |           |              |         |       |  |        |          |

ND = Mot detected at the report limit.
Matrix Spike Recovery For TCLP analytes reported From Natch AC
TCLP prepartion follows method 1311, SN-846 Revision 3.

All results reported on a net neight basis.



2960 Foster Creighton Dr Nashville, TN 37204 615-726-0177 Fax: 615-726-0954

#### ANALYTICAL REPORT

Laboratory Number: 00-A114303 Sample ID: MINERAL SAND CONCENTRATE

Page 2

These results relate only to the items tested.

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Report Approved By:

Report Date: 8/22/00

Paul E. Lane, Jr., Lab Director Michael H. Dunn, M.S., Technical Director Johnny A. Mitchell, Dir. Technical Serv. Eric S. Smith, Assistant Technical Director

Gail A. Lage, Technical Serv. Glenn L. Norton, Technical Serv. Kelly S. Comstock, Technical Serv. Pamela A. Langford, Technical Serv.

Laboratory Certification Number: 00323

ITEM 4



## FROEHLING & ROBERTSON, INC.

GEOTECHNICAL • ENVIRONMENTAL • MATERIALS ENGINEERS • LABORATORIES "OVER ONE HUNDRED YEARS OF SERVICE" Page 1 of 3

#### **CERTIFICATE OF ANALYSIS**

October 12, 2000

LAB#:

0009170

CLIENT:

Iluka Resources, Inc.

12472 St. John Church Road Stony Creek, VA 23882-0129

ATTN: Chee Saunders

PROJECT NAME:

**VPDES Stormwater** 

SAMPLED BY:

S. Conaway

LAB RECEIPT:

9/26/00, 1630

| PARAMETER        | ANALYSIS DATE/TIME | METHOD                      | ANALYST |
|------------------|--------------------|-----------------------------|---------|
| Silver           | 10/9/00, 1428      | EPA 200.7                   | MDW     |
| Alkalinity       | 10/10/00, 1100     | SM18/2320 B                 | BCD     |
| Ammonia-Nitrogen | 9/26/00, 1700      | SM18/4500-NH <sub>3</sub> F | BCD     |
| Arsenic          | 10/5/00, 1057      | EPA 200.7                   | MDW     |
| 3arium           | 10/5/00, 1057      | EPA 200.7                   | MDW     |
| BOD              | 9/27/00, 1350      | SM18/5210 B                 | AR/BCD  |
| Cadmium          | 10/6/00, 0846      | EPA 200.7                   | MDW     |
| COD              | 9/28/00, 1000      | HACH (SM18/5220 D)          | BCD     |
| Chromium         | 10/5/00, 1057      | EPA 200.7                   | MDW     |
| Metals Digest    | 9/29/00, 0940      | EPA 600/4-79/020            | DO      |
| Iron             | 10/5/00, 1057      | EPA 200.7                   | MDW     |
| Mercury          | 10/3/00, 0900      | EPA 245.1                   | RHS     |
| Potassium        | 10/10/00, 1503     | EPA 200.7                   | MDW     |
| Sodium           | 10/6/00, 1551      | EPA 200.7                   | MDW     |
| Nitrite-Nitrogen | 9/27/00, 1400      | SM18/4500-NO <sub>2</sub> B | BCD     |
| Nitrate-Nitrogen | 9/27/00, 1400      | SM18/4110 B                 | BCD     |
| Oil & Grease     | 9/28/00, 1430      | SM18/5520 B                 | DO/BCD  |
| Total Phosphorus | 10/4/00, 1100      | SM18/4500-P B&E             | AR      |
| Lead             | 10/5/00, 1057      | EPA 200.7                   | MDW     |
| Titanium         | 10/10/00, 1049     | EPA 200.7                   | MDW     |
| pН               | 9/26/00, 1615      | EPA 150.1                   | BCD     |

Continued on the following page

Audrey N. Brubeck

Jaboratory Manager ...

ANB/spt

CERTIFICATIONS

AIIIA ELLAP - 100533

VIRGINIA DRINKING WATER - 00150 NORTH CAROLINA DEHNR - 432

SOUTH CAROLINA DHEC - 93010001 & 93010002 MARYLAND DRINKING WATER - 279

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#### CERTIFICATE OF ANALYSIS, continued

October 11, 2000

LAB#:

0009170

CLIENT:

Iluka Resources, Inc.

12472 St. John Church Road Stony Creek, VA 23882-0129

ATTN: Chee Saunders

PROJECT NAME:

VPDES Stormwater

| PARAMETER              | ANALYSIS DATE/TIME | METHOD                                | ANALYST |
|------------------------|--------------------|---------------------------------------|---------|
| Selenium               | 10/5/00, 1057      | EPA 200.7                             | MDW     |
| Sulfate                | 9/27/00, 1400      | SM18/4110 B                           | BCD     |
| TKN                    | 9/27/00, 0800      | SM18/4500-N Org C & NH <sub>3</sub> E | AR      |
| TOC                    | 10/4/00, 0830      | SM18/5310 C                           | BCD     |
| Total Solids           | 9/26/00, 1700      | SM18/2540 B                           | BCD     |
| Total Suspended Solids | 10/2/00, 1000      | SM18/2540 D                           | AR      |

Results on the following page



Page 3 of 3 0009170 VPDES

Stormwater

Quant.

#### RESULTS:

F&R#:

0009170-1

SAMPLE ID:

Retention Pond #3

DATE/TIME:

9/26/00, 1245

MATRIX:

Water grab

|                              |      |     |  |       | Quant. |
|------------------------------|------|-----|--|-------|--------|
|                              |      |     |  |       | Limit: |
|                              | DOL  |     |  |       | 0.2 ,  |
| Silver, mg/L                 | BQL  |     |  |       | 0.2 ,  |
| Alkalinity, mg/L             | 15   |     |  |       |        |
| Ammonia-Nitrogen, mg/L       | 0.10 |     |  |       | 0.05   |
| Arsenic, mg/L                | BQL  |     |  |       | 0.2    |
| Barium, mg/L                 | BQL  |     |  |       | 0.1    |
| BOD, mg/L                    | 2    |     |  |       | 2      |
| Cadmium, mg/L                | BQL  |     |  |       | 0.2    |
| COD, mg/L                    | 37   |     |  |       | (30)   |
| Chromium, mg/L               | BQL  |     |  |       | 0.1    |
| Iron, mg/L                   | 1.9  |     |  |       | 0.2    |
| 'ercury, mg/L                | BQL  |     |  |       | 0.0005 |
| otassium, mg/L               | 3.4  |     |  |       | 1.0    |
| Sodium, mg/L                 | 11   |     |  |       | 1.0    |
| Nitrite-Nitrogen, mg/L       | BQL  |     |  |       | 0.05   |
| Nitrate-Nitrogen, mg/L       | 0.14 |     |  |       | 0.05   |
| Oil & Grease, mg/L           | BQL  |     |  |       | 5      |
| Total Phosphorus, mg/L       | 0.04 |     |  | r     | 0.02   |
| Lead, mg/L                   | BQL  |     |  |       | 0.2    |
| Selenium, mg/L               | BQL  |     |  |       | 0.4    |
| Sulfate, mg/L                | 2.8  |     |  |       | 0.5    |
| TKN, mg/L                    | 1.1  | (4) |  |       | 0.5    |
| TOC, mg/L                    | 9.7  |     |  |       | 0.5    |
| Total Solids, mg/L           | 112  |     |  |       | 10     |
| Total Suspended Solids, mg/L | 12   |     |  |       | . 1    |
| Titanium, mg/L               | BQL  |     |  |       | 0.5    |
|                              |      |     |  |       |        |
| pH, su                       | 6.4  |     |  | (4) 2 |        |
|                              |      |     |  |       |        |

## Primary Laboratories, Inc.

7423 Lee Davis Road • Mechanicsville, VA 23111 • Telephone (804) 559-9004 • Fax (804) 559-9306



## ANALYTICAL LABORATORY REPORT

29-Mar-07

ILUKA Resource, Inc. Attn: Kevin Rideout

12472 St. John Church Road Stony Creek, Virginia 23882

Date Received:

22-Mar-07

Date Sampled:

22-Mar-07

Work Order No:

0703207-01

Client ID:

MSP Permit Renewal Sampling-002

| Client ID:          | MSP Perm | it Renewal S | ampling-002 |                |           |          |          |
|---------------------|----------|--------------|-------------|----------------|-----------|----------|----------|
| Test                | Final    | Reporting    | Units of    | EPA Test       | Date      | Time     | Tech.    |
| Description         | Result   | Limit        | Measure     | Method         | Analyzed  | Analyzed | Initials |
| BOD                 | <2.0     | 2.0          | mg/L        | 405.1          | 23-Mar-07 | 15:30    | NA       |
| COD                 | 14.0     | 2.0          | mg/L        | 410.2          | 29-Mar-07 | 09:00    | NA       |
| тос                 | 6.31     | 1.00         | mg/L        | 415.1          | 29-Mar-07 | 07:30    | PB       |
| TPH-Diesel Range    | <1       | 1            | mg/L        | 3510/8015B (M) | 27-Mar-07 | 14:22    | HV       |
| TPH-Gasoline Range  | <1       | 1            | mg/L        | 5030/8015B (M) | 22-Mar-07 | 18:41    | HV       |
| T. Phosphorus       | 0.03     | 0.01         | mg/L        | 365.2          | 28-Mar-07 | 15:00    | NA       |
| T. Organic Nitrogen | <1.0     | 1.0          | mg/L        | 351.3          | 29-Mar-07 | 15:30    | NA       |
|                     |          |              |             |                |           |          |          |

Date Sampled:

22-Mar-07

Work Order No:

0703207-02

Client ID:

E. Coli - 002

| Test        | Final  | Reporting | Units of  | EPA Test | Date      | Time     | Tech.    |
|-------------|--------|-----------|-----------|----------|-----------|----------|----------|
| Description | Result | Limit     | Measure   | Method   | Analyzed  | Analyzed | Initials |
| E. Coli     | <2.0   | 2.0       | MPN/100ml | SM 9221F | 22-Mar-07 | 16:30    | AS       |

Signature:

Parry L. Bragg

Laboratory Manager

Date: 3-29-07

These analytical results are based upon materials provided by the client and are intended for the exclusive use of the client. These analytical results represent the best judgement of Primary Laboratories, Inc. Primary Laboratories, Inc. assumes no responsibility, express or implied, as to the interpretation of the analytical results contained in this report. This report is not to be reproduced except with the written approval of Primary Laboratories, Inc.

# APPENDIX 4 RENTION TIME CALCULATIONS FOR RETENTION POND B

# Flow from the 1 hour / 25 year storm event was calculated using the Rational Method:

 $Q = C \times i \times A$ 

Q = Flow (cfs)

C = Runoff Coefficient

i = Inches of rain produced by the 1 hour / 25 year storm event

A = Acres of drainage area

As described in the Virginia Department of Conservation and Recreation's *Virginia Erosion and Sediment Control Handbook*, Third Edition, 1992.

#### The following values were used in the calculation:

The runoff coefficient (**C**) for the site is estimated to be **0.05**, based on the Land Use classification of Light Industrial.

Rainfall expected from the 1 hour / 25 year storm event (i) is approximately **2.75 inches.** This is based on the average of the City of Richmond and Greensville County curve values obtained from the DCR ESC Handbook.

The area (A) served by Retention Pond B is approximately 11.85 acres.

Therefore:

Q = 0.05 x 2.75 in x 11.85 ac = 16.3 cubic feet per second ⇒ 978 cubic feet per minute ⇒ 7,316 gallons per minute 0.5 used

The volume of Retention Pond B is approximately **1.1 million gallons**.

Therefore, retention time for runoff produced by the 1 hour / 25 year storm is:

Retention Time = 1,100,000 gallons ÷ 7,316 gallons per minute = 150.4 minutes ⇒ **2.5 hours** 

#### Jenkins, Ray

From: Chamberlain, Corwin

Sent: Tuesday, September 04, 2007 11:01 AM

To: Jenkins, Ray

Subject: FW: VPDES information

This is additional info related to the dry site app.

Corwin Chamberlain

**Environmental Specialist** 

Virginia Dept. of Environmental Quality

804-527-5081

Fax 527-5106

----Original Message----

From: Allen, John [mailto:John.Allen@iluka.com]

Sent: Tuesday, August 14, 2007 5:40 PM

**To:** Chamberlain, Corwin **Subject:** VPDES information

Corey,

Per your letter dated August 7, 2007, I have compiled the attached information, which is to be incorporated into the renewal package for VPDES permit #VA0090981.

Please contact me if you have any questions regarding this information.

Cheers,

-John A. Allen

Environmental Officer Iluka Resources Inc

Office: (434)-348-4315

Cell: (804)-943-5611

john.allen@iluka.com

<<MSP VPDES Addendum etal.pdf>>

**NOTICE** - Mail attachments have been compressed automatically and appear as .ZIP files. Should you be unable to access compressed attachments free compression software (e.g FreeZip) is readily available from the Internet.

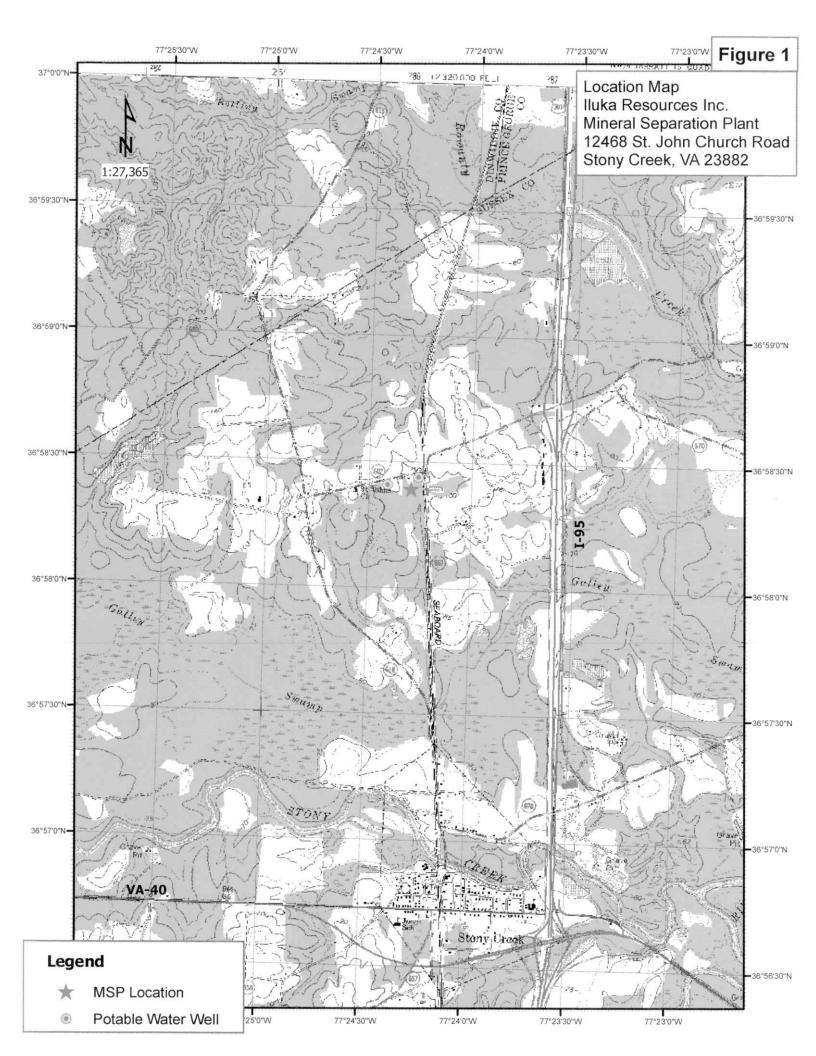
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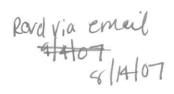
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|                      | Service of fire | 1000    |
|----------------------|-----------------|---------|
| rm Approved. OMB No. | I have good he  | AT 2017 |
| rm Approved. OMB No. | 2040-0086       | 01 2001 |

| Please print or t   | ype in the unshad                       | ed areas only.  |                    |         |                           |  | Form Approved. OMB No. 2040-0  | 086        | uc       | 01 20          |
|---|---|---|--------------------|---------|---------------------------|--|--|------------|----------|----------------|
| FORM  |   |   |                    |         |                           | ON AGENCY  | I. EPA I.D. NUMBER   | esta nor a | 100 H    |                |
| 1   | <b>\$EPA</b>                            |   |                    |         | FORMA<br>ermits Prog      |  | s<br>F VA0090981   | įv.        | - 5      | T/A C          |
| GENERAL   |   |   |                    |         |                           | ore starting.)   | 1 2  | - 12       | 143      | 15             |
| LABEL   | ITEMS                                   |   |                    |         |                           |  | GENERAL INSTRU   |            |          | t it in the    |
|   |   |   |                    |         |                           |  | designated space. Review the inform  | nation c   | arefully | ; if any of it |
| I. EPA I.D.   | NUMBER                                  |   |                    |         |                           |  | appropriate fill-in area below. Also, if   | any of     | the prej | printed data   |
| designated space. Review the information carefully; if any of the |   |   |                    |         |                           |  |  |            |          |                |
| V. FACILITY   | MAILING                                 |   |                    |         |                           |  | need not complete Items I, III, V, a   | and VI (   | (except  | VI-B which     |
| ADDRES  | S                                       |   |                    |         |                           |  | has been provided. Refer to the ins  | struction  | is for d | etailed item   |
| VI. FACILITY  | LOCATION                                |   |                    |         |                           |  |  | rization   | s under  | which this     |
| II. POLLUTANT   | CHARACTERIS                             | TICS  |                    |         | 198                       | <b>计算数据的</b>   |  |            |          | F-1866         |
| submit this form  | m and the supple<br>o" to each questio  | mental form listed in the pare<br>in, you need not submit any o | nthesis<br>f these | forms   | wing the qu<br>s. You may | estion. Mark "X" in the box in<br>answer "no" if your activity is  | n the third column if the supplemen  | ntal for   | m is a   | ttached. If    |
|   |   |   | VED                |         |                           |  |  | VEC        |          |                |
|   | SPECIFIC QL                             | JESTIONS  | TES                | NO      |                           | SPECIF   | IC QUESTIONS   | 150        | NO       |                |
|   |   |   |                    |         |                           | include a concentrate<br>aquatic animal produ  | d animal feeding operation or<br>ction facility which results in a   |            |          | 21             |
| C la this a fee   | ility which ourron                      | tly recults in discharges to                                    |                    | 17      | 300.00                    |  |  |            | 200      | 21             |
| waters of t   | he U.S. other tha                       |   | X                  |         | X                         | or B above) which will r   | esult in a discharge to waters of  |            | X        |                |
| ,   | (6)                                     |   | 22                 | 23      | 24                        |  |  | 25         | 26       | 27             |
|   |   |   |                    | Y       |                           |  |  |            | v        |                |
|   | ,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,, |   |                    | •••     |                           |  |  |            | 1 50     |                |
| C. Do vou or w  | ill you inject at thi                   | a facility any produced water                                   | 28                 | 29      | 30                        |  |  | -          | 32       | 33             |
| or other flu  | uids which are                          | brought to the surface in                                       |                    |         |                           | processes such as minir  | ng of sulfur by the Frasch process,  |            |          |                |
|   |   | oil or natural gas production,<br>ed recovery of oil or natural |                    | X       |                           |  | erals, in situ combustion of fossil<br>hermal energy? (FORM 4)   |            | X        |                |
| gas, or inje  |   | age of liquid hydrocarbons?                                     |                    |         |                           | 1000, 00 1000, 700, 700, 700, 700, 700,  | 37 (   |            |          |                |
| (FORM 4)  |   | •   | 34                 | 35      | 36                        | A DE SENS VINNESSES DE SESSES  | AND THE PERSON AND TH | 37         | 38       | 39             |
|   |   | tionary source which is one<br>listed in the instructions and   |                    | x       |                           |  | sed stationary source which is<br>adustrial categories listed in the   |            | v        |                |
| which will p  | otentially emit 10                      | 00 tons per year of any air<br>Clean Air Act and may affect     |                    | ^       |                           |  | will potentially emit 250 tons per<br>regulated under the Clean Air Act  |            | X        |                |
|   |   | t area? (FORM 5)  | 40                 | 41      | 42                        |  | located in an attainment area?   |            | 44       | 45             |
| III. NAME OF  | FACILITY                                |   |                    |         |                           | (i oran o)   |  |            |          | Mary I.        |
| c   |   |   | Ţ                  |         | TIL                       | The transfer of the transfer o |  | 1          |          |                |
| 1 -   | luka keso                               | urces INC. Mines  | raı                | Sep     | aratio                    | on Plant (MSP)   |  |            |          |                |
| 15   16 - 29   30   IV. FACILITY                                  | CONTACT                                 |   |                    | _       |                           |  |  | 69         |          |                |
| IV. FACILITI  | CONTACT                                 | A. NAME & TITLE (last   | first              | & title | 10                        |  | B. PHONE (area code & no.)   |            |          |                |
| 0 0 7   |   |   |                    | TT      |                           |  | (434) 348-4302   |            | 7000     |                |
| 2 Sale,   | Allan Pr                                | esident   |                    |         |                           | 45   |  | 55         |          |                |
| V FACILTY MA  | AILING ADDRESS                          |   |                    |         |                           |  | 46 48 49 51 52-  | 200        |          | A              |
| V.I AGILIT W  | ILLING ADDINES                          | A. STREET OR P  | .O. BO             | X       |                           |  |  |            | 416      |                |
| c 12472   | St. John                                | Chunch Dd   |                    |         |                           |  |  |            |          |                |
| 3 12472   | St. John                                | Church Ru   |                    |         |                           | 45   |  |            |          |                |
| 15 16   |   | B. CITY OR TOWN   |                    |         |                           | C. STATE   | D. ZIP CODE  |            | _        |                |
| 6 0 1   |   |   |                    |         |                           |  |  |            |          |                |
| 4 Stony   | Creek                                   |   |                    |         |                           |  | 23882  |            |          |                |
| VI. FACILITY  | LOCATION                                |   | 575Y               |         | SCHOOL                    | 40 41 42   |  | 65.6       |          |                |
| VI. TAOLETT   | 0.000                                   | REET, ROUTE NO. OR OTHE   | R SPE              | CIFIC   | DENTIFI                   | ER .   |  |            |          |                |
| 12469   | C+ Tohn                                 | Church Pd   | 1 1                |         |                           |  |  |            |          |                |
| 3   | JU. UUIIII                              | Church Rd.  |                    |         |                           | 45   | Lay Jr Trus  |            |          |                |
| 15 16   |   | B. COUNTY   | / NAM              | E       |                           |  |  |            |          |                |
| Sugger  |   |   |                    |         |                           |  |  |            |          |                |
| Sussex  |   |   |                    |         |                           |  | 70   |            |          |                |
|   |   | C. CITY OR TOWN   |                    |         |                           | D. STATE   | E. ZIP CODE F. COUNTY C  | ODE (      | if know  | n)             |
| 6 Stony   | Creek                                   |   | J.                 |         | 1 1 1                     | VA VA  | 23882 0183   | 3          |          |                |
| 15 16   |   |   |                    |         |                           | 40 41 42   | 47 51 52   | -54        |          |                |

| CONTINUED FROM THE FRONT   |  |
|--|--|
| VII. SIC CODES (4-digit, in order of priority)   | P. OFFICIAL PROPERTY OF THE PR |
| A. FIRST  (specify) Miscellaneous Metal Ores, Not Elsewhere Classified.  | B. SECOND  |
| 7 1099 The plant produces zircon and ilmentite sand products from sedimentary ore deposits via gravity and electrostatic   | 7 na   |
| 15 16 - 19 separation methods C. THIRD   | 15 16 · 19   |
| c     (specify) na   | D. FOURTH  |
| 7 na   | 7 na (spesy)   |
| 15 16 · 19   | 15 16 - 19   |
| VIII. OPERATOR INFORMATION  A. NAME  | B.Is the name listed in Item   |
|  | VIII-A also the owner?   |
| 8 Iluka Resources INC.   | ☑ YES ☐ NO   |
| 15 16  |  |
| C. STATUS OF OPERATOR (Enter the appropriate letter into the   |  |
| S = STATE M = PUBLIC (other than federal or state) P   | pased in Perth and traded on the Australian (434) 348-4300   |
| O = OIHER (specify)  | Recurities Exchange (ASX). US Operations are represented by the Stony Creek office.  |
| 1 77 1   | 13 0 - 10 19 - 21 22 - 20  |
| E. STREET OR P.O. BOX  |  |
| 12472 St. John Church Rd   |  |
| 26   | 65   |
| F. CITY OR TOWN  | G. STATE   H. ZIP CODE   IX. INDIAN LAND   |
| Ctony Grook  | VA 23882 Street  |
| B Stony Creek  | 52   |
| 15 16  | 40 41 42 47 - 51   |
| X. EXISTING ENVIRONMENTAL PERMITS  A. NPDES (Discharges to Surface Water)  D. PSD (Air E   | missions from Proposed Sources)  |
| C T I C T I  | Missions from Froposed sources)  |
| 9 N VA0090981 9 P na   |  |
| 15 16 17 18 30 15 16 17 18   | 30   |
| B. UIC (Underground Injection of Fluids)   | E. OTHER (specify)   |
| g U na g NSR Re  | g.# 51220 (specify) Virginia new source review permit for stationary minor sources of air  |
| 15 16 17 18 30 15 16 17 18   | emissions.   |
| C. RCRA (Hazardous Wastes)   | E. OTHER (specify)   |
| o D NARO51   | (specify) VPDES governing discharges from the MSP's Sediment Pond and the  |
| 9 7  | Administration Building's Sediment Pond.   |
| 15 16 17 18 30 15 16 17 18 XI. MAP   | 30   |
|  | e mile beyond property boundaries. The map must show the outline of the facility, the  |
|  | of its hazardous waste treatment, storage, or disposal facilities, and each well where it  |
| injects fluids underground. Include all springs, rivers, and other surface water bodies  | s in the map area. See instructions for precise requirements. See Appendix 1, Figures 1 and 2  |
| XII. NATURE OF BUSINESS (provide a brief description)  |  |
| Iluka Resoucres Inc. is a primary producer of zircon ar  |  |
| various industries engaged in the production of investmingredients. Operations at the Stony Creek Mineral Sepa   |  |
| plant feed, material drying, ilmenite separtation, ziro  |  |
| treatment, product storage and loadout, tailings storage   |  |
| equipment fueling and maintenance, and groundwater mana  | gement.  |
|  |  |
|  |  |
|  |  |
|  |  |
|  |  |
|  |  |
|  | *  |
|  |  |
|  |  |
|  |  |
| XIII. CERTIFICATION (see instructions)   |  |
| The second secon | the information submitted in this application and all attachments and that, based on my  |
| inquiry of those persons immediately responsible for obtaining the information cont  | ained in the application, I believe that the information is true, accurate, and complete. I  |
| am aware that there are significant penalties for submitting false information, includi-   |  |
| A. NAME & OFFICIAL TITLE (type or print)  B. SIGNATUR  | C. DATE SIGNED   |
| ALLAN R SALE PRESIDENT   | 12hle 7/25/67  |
|  | 7/20/07  |
| COMMENTS FOR OFFICIAL USE ONLY   | ed a trabal a specific production and the second  |
| c  |  |
| c  |  |





#### VPDES Permit Application Addendum

| Entity to whom the permit is to be issued:     Iluka Resources Inc.  |
|--|
| Who will be legally responsible for the wastewater treatment facilities and compliance with the permit? This may or may not be the facility or property owner.   |
| 2. Is this facility located within city or town boundaries? Y / №  |
| 3. What is the tax map parcel number for the land where this facility is located? 48-A-16B   |
| 4. For the facility to be covered by this permit, how many acres will be disturbed during the next five years due to new construction activities? 0  |
| 5. ALL FACILITIES: What is the design average flow of this facility? 0.072 MGD Industrial facilities: What is the max. 30-day avg. production level (include units)? 29.4 gpm  |
| In addition to the above design flow or production level, should the permit be written with limits for any other discharge flow tiers or production levels?  |
| If "Yes", please specify the other flow tiers (in MGD) or production levels:   |
| Please consider: Is your facility's design flow considerably greater than your current flow? Do you plan to expand operations during the next five years?  |
| 6. Nature of operations generating wastewater:  Removal of iron and other impurities from the surfaces of the zircon sand product using sulfuric acid  |
| % of flow from domestic connections/sources  Number of private residences to be served by the wastewater treatment facilities:   |
| 100 % of flow from non-domestic connections/sources  |
| 7. <b>Mode of discharge</b> :Continuous <b>X</b> IntermittentSeasonal Describe frequency and duration of intermittent or seasonal discharges:Approximately 1 discharge per week, typically less than 24 hours in duration  |
| 8. Identify the characteristics of the receiving stream at the point just above the facility's discharge point:  Permanent stream, never dry Intermittent stream, usually flowing, sometimes dry Ephemeral stream, wet-weather flow, often dry Effluent-dependent stream, usually or always dry Lake or pond at or below the discharge point Other: Swamp, sometimes dry |
| 9. Approval Date(s): O & M Manual Sludge/Solids Management Plan 1/27/2003  |
| Have there been any changes in your operations or procedures since the above approval dates? Y / ₩   |
| 10. What is the average hardness of the effluent that you discharge? 1829 mg/L   |

CONTINUED FROM THE FRONT C. Except for storm runoff, leaks, or spills, are any of the discharges described in Items II-A or B intermittent or seasonal? ✓ YES (complete the following table) NO (go to Section III) 3. FREQUENCY 4. FLOW a. DAYS PER B. TOTAL VOLUME a. FLOW RATE (in mgd) 2. OPERATION(s) CONTRIBUTING FLOW WEEK b. MONTHS PER YEAR 1. OUTFALL NUMBER (list) (specify average) 1. LONG TERM AVERAGE DURATION (in days) 2. MAXIMUM 1. LONG TERM 2. MAXIMUM (list) (specify average) DAILY DAILY 001 Zircon Finishing Plant Effluent 50,400 gpd .04234 .05040 42,444 gpd (ZFP) Treatment Plant (ETP) average average III. PRODUCTION A. Does an effluent guideline limitation promulgated by EPA under Section 304 of the Clean Water Act apply to your facility? YES (complete Item III-B) NO (go to Section IV) B. Are the limitations in the applicable effluent guideline expressed in terms of production (or other measure of operation)? YES (complete Item III-C) NO (go to Section IV) C. If you answered "yes" to Item III-B, list the quantity which represents an actual measurement of your level of production, expressed in the terms and units used in the applicable effluent guideline, and indicate the affected outfalls. 1. AVERAGE DAILY PRODUCTION 2. AFFECTED OUTFALLS c. OPERATION, PRODUCT, MATERIAL, ETC. a. QUANTITY PER DAY b. UNITS OF MEASURE (list outfall numbers) (specify) na na na IV. IMPROVEMENTS A. Are you now required by any Federal, State or local authority to meet any implementation schedule for the construction, upgrading or operations of wastewater treatment equipment or practices or any other environmental programs which may affect the discharges described in this application? This includes, but is not limited to, permit conditions, administrative or enforcement orders, enforcement compliance schedule letters, stipulations, court orders, and grant or loan conditions. YES (complete the following table) NO (go to Item IV-B) 1. IDENTIFICATION OF CONDITION, 2. AFFECTED OUTFALLS 4. FINAL COMPLIANCE DATE 3. BRIEF DESCRIPTION OF PROJECT AGREEMENT, ETC. b. SOURCE OF DISCHARGE a. NO. a. REQUIRED b. PROJECTED na na na na

| B. | discharges) you now have underwa | y or which yo | describing any additional ou plan. Indicate whether ea | water pollution control programs (or other environmenta ach program is now underway or planned, and indicate you DGRAMS IS ATTACHED. | I projects which<br>ur actual or plann | may affect your<br>ed schedules for |
|----|----------------------------------|---------------|--|--|--|-------------------------------------|

PLEASE PRINT OR TYPE IN THE UNSHADED AREAS ONLY. You may report some or all of this information on separate sheets (use the same format) instead of completing these pages. SEE INSTRUCTIONS.

OUTFALL NO. 001 PART A -You must provide the results of at least one analysis for every pollutant in this table. Complete one table for each outfall. See instructions for additional details. EPA I.D. NUMBER (copy from Item 1 of Form 1) VA0090981 V. INTAKE AND EFFLUENT CHARACTERISTICS (continued from page 3 of Form 2-C)

|                                       |                        |                 |  | 2. EFFLUENT     | ENT                                     |          |                       | 3. UNITS (specify if blank) | TS<br>Slank) | 4                             | 4. INTAKE (optional) |                       |
|---------------------------------------|------------------------|-----------------|--|-----------------|---|----------|-----------------------|-----------------------------|--------------|-------------------------------|----------------------|-----------------------|
|                                       | a. MAXIMUM DAILY VALUE | AILY VALUE      | b. MAXIMUM 30 DAY VALUE (if available) | DAY VALUE       | c. LONG TERM AVRG. VALUE (if available) | G. VALUE |                       |                             |              | a. LONG TERM<br>AVERAGE VALUE | ERM<br>/ALUE         | 1                     |
| 1. POLLUTANT                          | (1)<br>CONCENTRATION   | (2) MASS        | (1)<br>CONCENTRATION                   | (2) MASS        | (1) CONCENTRATION                       | (2) MASS | d. NO. OF<br>ANALYSES | a. CONCENTRATION            | b. MASS      | (1)<br>CONCENTRATION          | (2) MASS             | b. NO. OF<br>ANALYSES |
| a. Biochemical Oxygen<br>Demand (BOD) | <3.0                   |                 |  |                 | J.                                      |          | П                     | mg/L                        |              |                               |                      |                       |
| b. Chemical Oxygen<br>Demand (COD)    | 22.1                   |                 |  |                 |   |          | П                     | mg/L                        |              |                               |                      |                       |
| c. Total Organic Carbon (TOC)         | 5.38                   |                 |  |                 |   |          | 1                     | mg/L                        |              |                               |                      |                       |
| d. Total Suspended<br>Solids (TSS)    | 52.8                   |                 |  |                 | 19.06                                   |          | 7                     | mg/L                        |              |                               |                      |                       |
| e. Ammonia (as N)                     | 0.27                   |                 |  |                 | 0.115                                   |          | 4                     | mg/L                        |              |                               |                      |                       |
| f. Flow                               | VALUE 35 gpm           | md              | VALUE                                  |                 | VALUE 29.4 gpm                          | ш        | 14                    |                             |              | VALUE                         |                      |                       |
| g. Temperature (winter)               | VALUE 7.3              |                 | VALUE                                  |                 | VALUE                                   |          | 19                    | ပ့                          |              | VALUE                         |                      |                       |
| h. Temperature<br>(summer)            | VALUE 35.5             | 22              | VALUE                                  |                 | VALUE                                   |          | 80                    | ပ္                          |              | VALUE                         |                      |                       |
| Hd :                                  | MINIMUM<br>6.15        | MAXIMUM<br>7.06 | MINIMUM<br>6.02                        | MAXIMUM<br>8.96 |   |          | 57                    | STANDARD UNITS              | UNITS        |                               |                      |                       |
| C H                                   |                        |                 |  |                 | 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1   |          |                       |                             | 1            |                               |                      |                       |

PART B — Mark "X" in column 2-a for each pollutant you know or have reason to believe is present. Mark "X" in column 2-b for each pollutant you believe to be absent. If you mark column 2a for any pollutant which is limited either adjusted by the expressity, in an effluent limitations guideline, you must provide the results of at least one analysis for that pollutant. For other pollutants for which you mark column 2a, you must provide the instructions for additional details and requirements.

| anh                            | אוווומוואב ממו      | מ כו מון כעם | qualiticative data of all explanation of their presence in your disordings. Complete one table to each outsit. | a in a in a in a | ischarge, complete               | olle table loi    | cacil oduali. See ale   | in su dedonis io | additional des        | illo alla ledallell | CINO.   |                               |                      |                       |
|--------------------------------|---------------------|--------------|--|------------------|----------------------------------|-------------------|---|------------------|-----------------------|---------------------|---------|-------------------------------|----------------------|-----------------------|
|                                |                     | 2. MARK "X"  |  |                  | 3.                               | 3. EFFLUENT       |   |                  |                       | 4. UNITS            | -S      | 5. INT/                       | 5. INTAKE (optional) | )                     |
| 1. POLLUTANT<br>AND            |                     |              | a. MAXIMUM DAILY VALUE   | ILY VALUE        | b. MAXIMUM 30 DAY (if available) | DAY VALUE<br>ble) | b. MAXIMUM 30 DAY VALUE c. LONG TERM AVRG. VALUE (if available) | VRG. VALUE       |                       | 1                   |         | a. LONG TERM AVERAGE<br>VALUE | VERAGE               | (                     |
| (if available)                 | BELIEVED<br>PRESENT | BELIEVED     | (1)<br>CONCENTRATION   | (2) MASS         | (1)<br>CONCENTRATION             | (2) MASS          | (1)<br>CONCENTRATION  | (2) MASS         | d. NO. OF<br>ANALYSES | a. CONCENTRATION    | b. MASS | b. MASS CONCENTRATION         | (2) MASS             | b. NO. OF<br>ANALYSES |
| a. Bromide<br>(24959-67-9)     |                     | ×            |  |                  |                                  |                   |   |                  |                       |                     |         |                               |                      |                       |
| b. Chlorine, Total<br>Residual |                     | ×            |  |                  |                                  |                   |   |                  |                       |                     |         |                               |                      |                       |
| c. Color                       |                     | ×            |  |                  |                                  |                   |   |                  |                       |                     |         |                               |                      |                       |
| d. Fecal Coliform              | ×                   |              | 2  |                  |                                  |                   |   |                  | Н                     | MPN/100mL           |         |                               |                      |                       |
| e. Fluoride<br>(16984-48-8)    |                     | ×            |  |                  |                                  |                   |   |                  |                       |                     |         |                               |                      |                       |
| f. Nitrate-Nitrite (as N)      |                     | ×            |  |                  |                                  |                   |   |                  |                       | mg/L                |         |                               |                      |                       |

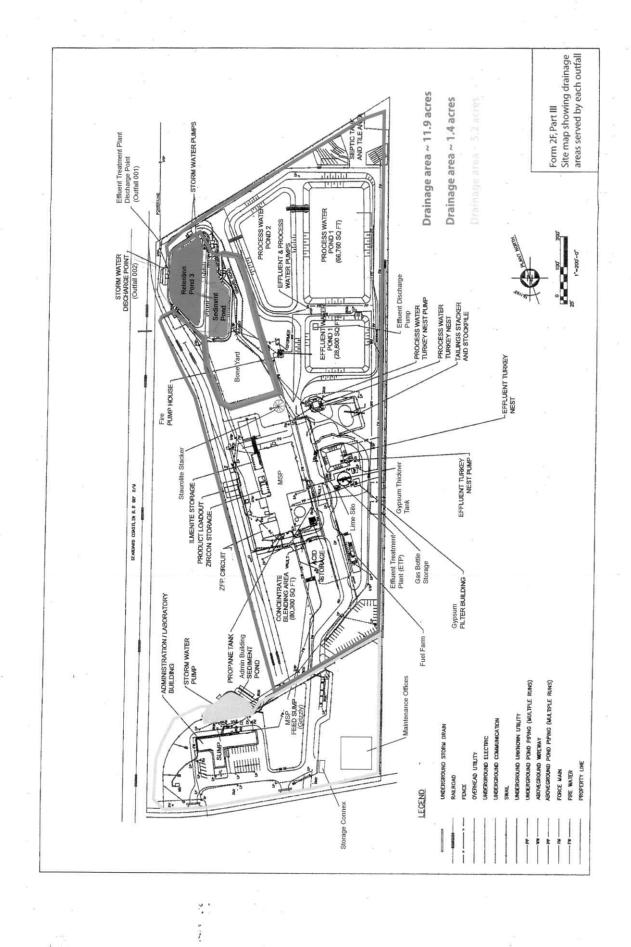
EPA Form 3510-2C (8-90)

PAGE V-1

CONTINUE ON REVERSE

ITEM V-B CONTINUED FROM FRONT

| TEM SECONDINATE INCIDENTAL PROPERTY OF THE PRO | 2           |          |                        |            |  |                   |   |          |                       |                  |         |                               |                      |                       |
|--|-------------|----------|------------------------|------------|--|-------------------|---|----------|-----------------------|------------------|---------|-------------------------------|----------------------|-----------------------|
| TNATILIDA  | 2. MARK "X" | <br>X    |                        |            | 3.                                     | 3. EFFLUENT       |   |          |                       | 4. UNITS         | TS      | 5. INTA                       | 5. INTAKE (optional) |                       |
|  | œ           | نم       | a. MAXIMUM DAILY VALUE | AILY VALUE | b. MAXIMUM 30 DAY VALUE (if available) | DAY VALUE<br>ble) | c. LONG TERM AVRG. VALUE (if available) |          |                       |                  |         | a. LONG TERM<br>AVERAGE VALUE |                      |                       |
| (if available)   | PRESENT     | BELIEVED | (1)<br>CONCENTRATION   | (2) MASS   | (1)<br>CONCENTRATION                   | (2) MASS          | (1)<br>CONCENTRATION                    | (2) MASS | d. NO. OF<br>ANALYSES | a. CONCENTRATION | b. MASS | (1)<br>CONCENTRATION          | ASS                  | b. NO. OF<br>ANALYSES |
| g. Nitrogen,<br>Total Organic (as  |             | ×        |                        |            |  |                   |   |          |                       |                  |         |                               |                      |                       |
| h, Oil and<br>Grease   |             | ×        |                        |            |  |                   |   |          |                       |                  |         |                               |                      |                       |
| i. Phosphorus<br>(as P), Total<br>(7723-14-0)  |             | ×        |                        |            |  |                   |   |          |                       |                  |         |                               |                      |                       |
| j. Radioactivity   |             |          |                        |            |  |                   |   |          |                       |                  |         |                               |                      |                       |
| (1) Alpha, Total   | ×           |          | 6.0 ± 0.7              |            |  |                   |   |          | Т                     | pCi/L            |         |                               |                      |                       |
| (2) Beta, Total  | ×           |          | 21 ± 3                 |            |  |                   |   |          | п                     | pCi/L            |         |                               |                      |                       |
| (3) Radium,<br>Total   |             | ×        |                        |            |  |                   |   |          |                       |                  |         |                               |                      |                       |
| (4) Radium 226,<br>Total   |             | ×        |                        |            |  |                   |   |          |                       |                  |         |                               |                      |                       |
| k. Sulfate<br>(as SO.)<br>(14808-79-8)   | ×           |          | 2635                   |            |  |                   |   |          | 23                    | mg/L             |         |                               |                      |                       |
| I. Sulfide (as S)  |             | ×        |                        |            |  |                   |   |          |                       |                  |         |                               |                      |                       |
| m. Sulfite<br>(as SO <sub>3</sub> )<br>(14265-45-3)  |             | ×        |                        |            |  |                   |   |          |                       |                  |         |                               |                      |                       |
| n. Surfactants   |             | ×        |                        |            |  |                   |   |          |                       |                  |         |                               |                      |                       |
| o. Aluminum,<br>Total<br>(7429-90-5)   |             | ×        |                        |            |  |                   |   |          |                       |                  |         |                               |                      |                       |
| p. Barium, Total<br>(7440-39-3)  |             | ×        |                        |            |  |                   |   |          |                       |                  |         |                               |                      |                       |
| q. Boron, Total<br>(7440-42-8)   |             | ×        |                        |            |  |                   |   |          |                       |                  |         |                               |                      |                       |
| r. Cobalt, Total<br>(7440-48-4)  |             | ×        |                        |            |  |                   |   |          |                       |                  |         |                               |                      |                       |
| s. Iron, Total<br>(7439-89-6)  | ×           |          | 0.364                  |            |  |                   |   |          | 2                     | mg/L             |         |                               |                      |                       |
| t. Magnesium,<br>Total<br>(7439-95-4)  |             | ×        |                        |            |  |                   |   |          |                       |                  |         |                               |                      |                       |
| u. Molybdenum,<br>Total<br>(7439-98-7)   |             | ×        |                        |            |  |                   |   |          |                       |                  |         |                               |                      |                       |
| v. Manganese,<br>Total<br>(7439-96-5)  |             | ×        |                        |            |  |                   |   |          |                       |                  |         |                               |                      |                       |
| w. Tin, Total<br>(7440-31-5)   |             | ×        |                        |            |  |                   |   |          |                       |                  |         |                               |                      |                       |
| x. Titanium,<br>Total<br>(7440-32-6)   |             | ×        |                        |            |  |                   |   |          |                       |                  |         |                               |                      |                       |
| EPA Form 3510-2C (8-90)  | C (8-90)    |          |                        |            |  |                   | PAGE V-2                                |          |                       |                  |         | CO                            | CONTINUE ON PAGE V-3 | PAGE V-3              |



Form Approved. OMB No. 2040-0086 Approval expires 5-31-92

#### VII. Discharge information (Continued from page 3 of Form 2F)

Part A - You must provide the results of at least one analysis for every pollutant in this table. Complete one table for each outfall. See instructions for additional details.

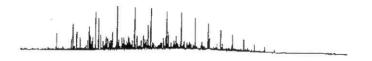
|  | 1000 (000)   | um Values<br>ude units)    |  | erage Values<br>eclude units) | Number                           |                                     |
|--|--|----------------------------|--|-------------------------------|----------------------------------|-------------------------------------|
| Pollutant<br>and<br>CAS Number<br>(if available) | Grab Sample<br>Taken During<br>First 20<br>Minutes | Flow-Weighted<br>Composite | Grab Sample<br>Taken During<br>First 20<br>Minutes | Flow-Weighted<br>Composite    | of<br>Storm<br>Events<br>Sampled | Sources of Pollutants               |
| Oil and Grease                                   | BQL mg/L   | N/A                        | na   | na                            | 2                                | na                                  |
| Biological Oxygen<br>Demand (BOD5)               | <2.0 mg/L  | na                         | na   | na                            | 2                                | Unknown                             |
| Chemical Oxygen<br>Demand (COD)                  | 37 mg/L  | na                         | 25.5 mg/L  | na                            | 2                                | Unknown                             |
| Total Suspended<br>Solids (TSS)                  | 42.4 mg/L  | na                         | 11.0 mg/L  | na                            | 16                               | Entrained solide in stormwater flow |
| Total Nitrogen                                   | 1.1 mg/L   | na                         | <1.1 mg/L  | na                            | 2                                | Unknown                             |
| Total Phosphorus                                 | 0.04 mg/L  | na                         | .035 mg/L  | na                            | 2                                | Unknown                             |
| рН   | Minimum 5.10                                       | Maximum 8.54               | Minimum  | Maximum                       | 95                               | na                                  |

Part B – List each pollutant that is limited in an effluent guideline which the facility is subject to or any pollutant listed in the facility's NPDES permit for its process wastewater (if the facility is operating under an existing NPDES permit). Complete one table for each outfall. See the instructions for additional details and requirements.

|  | (inclue  | ım Values<br>de units)     | Aver:  | age Values<br>lude units)  | Number                           |                       |
|--|--|----------------------------|--|----------------------------|----------------------------------|-----------------------|
| Pollutant<br>and<br>CAS Number<br>(if available) | Grab Sample<br>Taken During<br>First 20<br>Minutes | Flow-Weighted<br>Composite | Grab Sample<br>Taken During<br>First 20<br>Minutes | Flow-Weighted<br>Composite | of<br>Storm<br>Events<br>Sampled | Sources of Pollutants |
|  |  |                            |  |                            |                                  |                       |
|  |  |                            |  |                            |                                  |                       |
|  |  |                            |  |                            |                                  |                       |
| -  |  |                            |  |                            |                                  |                       |
|  |  |                            |  |                            |                                  |                       |
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|  |  |                            |  |                            |                                  |                       |
|  |  |                            |  |                            |                                  |                       |
|  |  |                            |  |                            |                                  | 11.1                  |
|  |  |                            |  |                            |                                  |                       |
|  |  |                            |  |                            |                                  |                       |
|  |  |                            |  |                            |                                  |                       |
|  |  |                            |  |                            |                                  |                       |
|  |  |                            |  |                            |                                  |                       |
|  |  |                            |  |                            |                                  |                       |
|  |  |                            |  |                            |                                  |                       |
|  |  |                            |  |                            |                                  |                       |
|  |  |                            |  |                            |                                  |                       |
|  |  |                            |  |                            |                                  |                       |

# Primary Laboratories, Inc.

2087 Dabney Road • Richmond, VA 23230 • Telephone (804) 213-0831 • Fax (804) 213-0842



# ANALYTICAL LABORATORY REPORT

04-Sep-03

ILUKA Resource, Inc. Attn: Chee Saunders 12472 St. John Church Stony Creek, Virginia 23882

Date Received:

28-Aug-03

Date Sampled:

27-Aug-03

Work Order No:

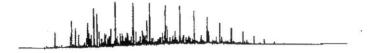
0308112-01

| Client ID:           | Outfall 001     |                    |                              |                    |                  |       |
|----------------------|-----------------|--------------------|------------------------------|--------------------|------------------|-------|
| Test<br>Description  | Final<br>Result | Reporting<br>Limit | Units of<br>Measure          | EPA Test<br>Method | Date<br>Analyzed | Tech. |
| Nitrate              | <0.01           | 0.01               | mg/L                         | 353.3              | 03-Sep-03        |       |
| Chloride             | 17.5            | 0.1                | mg/L                         | 325.3              | 02-Sep-03        | NA    |
| Sulfate              | 1,775           | 100                | mg/L                         | 375.4              | 04-Sep-03        | NA    |
| Hardness             | 1,635           | 1,0                | mg/L<br>as CaCO <sub>3</sub> | 130.2              | 02-Sep-03        | NA    |
| TSS                  | 4.0             | 0.5                | mg/L                         | 160.2              | 28-Aug-03        | н∨    |
| Specific Conductance | 2.37            | 0.2                | um/hos                       | 9050               | 03-Sep-03        | СН    |
| рH                   | 8.22            | -                  | pH Units                     | 150.1              | 28-Aug-03        | н٧    |
| Dissolved Chrome VI  | <0.005          | 0.005              | mg/L                         | SM3500             | 28-Aug-03        | NA    |
| Ammonia              | <0.01           | 0.01               | mg/L                         | 350.3              | 28-Aug-03        | NA    |
| Bicarbonate          | 14.0            | 0.1                | mg/L<br>as CaCO <sub>3</sub> | SM2320             | 4-Sep-03         | NA    |

NOTE: pH Temperture at 9.6 Degrees C.

## Primary Laboratories, Inc.

2087 Dabney Road • Richmond, VA 23230 • Telephone (804) 213-0831 • Fax (804) 213-0842



# ANALYTICAL LABORATORY REPORT

21-Nov-03

ILUKA Resource, Inc. Attn: Chee Saunders 12472 St. John Church Stony Creek, Virginia 23882

Date Received:

13-Nov-03

Project:

P.D.& J Environmental

Date Sampled:

13-Nov-03 0311078-01

Work Order No:

| Client ID:            | Effluent 001 |           |                              | Color Color |           |          |
|-----------------------|--------------|-----------|------------------------------|-------------|-----------|----------|
| Test                  | Final        | Reporting | Units of                     | EPA Test    | Date      | Tech.    |
| Description           | Result       | Limit     | Measure                      | Method      | Analyzed  | Initials |
| рН                    | 7.40         | -         | pH Units                     | 150.1       | 19-Nov-03 | HV       |
| TSS                   | 30,0         | 0.5       | mg/L                         | 160.2       | 20-Nov-03 | HV       |
| Chloride              | 15.3         | 0,1       | mg/L.                        | 325.3       | 17-Nov-03 | NA       |
| Specific Conductivity | 3230         | 0.2       | umhos/cm                     | 9050        | 19-Nov-03 | СН       |
| Sulfate               | 2,635        | 100       | mg/L                         | 375.4       | 18-Nov-03 | NA       |
| Hardness              | 2,471        | 5.0       | mg/L<br>as CaCO <sub>3</sub> | 130.2       | 19-Nov-03 | NA       |
| Ammonia               | 0.16         | 0.01      | mg/L                         | 350,3       | 20-Nov-03 | NA       |
| Alkalinity            | 12.3         | 0.1       | mg/L<br>as CaCO <sub>3</sub> | 310.1       | 17-Nov-03 | NA       |
| Dissolved Chrome VI   | <0.005       | 0.005     | mg/L                         | SM3500      | 14-Nov-03 | NA       |

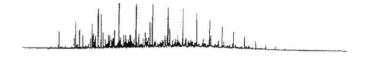
pH Temperature = 9.6 Degrees C.

14-Aug-07

Previously Reported 5/8/07

# Primary Laboratories, Inc.

7423 Lee Davis Road • Mechanicsville, VA 23111 • Telephone (804) 559-9004 • Fax (804) 559-9306



## ANALYTICAL LABORATORY REPORT

ILUKA Resource, Inc. Attn: Kevin Rideout

12472 St. John Church Road Stony Creek, Virginia 23882

Date Received:

4-May-07

Date Sampled:

3-May-07 0705038-01

Work Order No:

MSP Outfall no:

| Citeff (D.          | MSP Outfall     | 002                |                     |                          |                  |                  |                   |
|---------------------|-----------------|--------------------|---------------------|--------------------------|------------------|------------------|-------------------|
| Test<br>Description | Final<br>Result | Reporting<br>Limit | Units of<br>Measure | Standard<br>Methods (18) | Date<br>Analyzed | Time<br>Analyzed | Tech.<br>Initials |
| TSS                 | 5.4             | 1.0                | mg/L                | 2540 D                   | 7-May-07         | 12:00            | AS                |
| Hardness            | 500.1           | 1.0                | mg/L                | 2340 B                   | 14-Aug-07        | 13:00            | NA                |

Signature:

Parry L. Bragg

Laboratory Manager

Date: 8-14-07

These analytical results are based upon materials provided by the client and are Intended for the exclusive use of the client. These analytical results represent the best judgement of Primary Laboratories, Inc. Primary Laboratories, Inc. assumes no responsibility, express or implied, as to the interpretation of the analytical results contained in this report. This report is not to be reproduced except with the written approval of Primary Laboratories, Inc.

#### Bauer, Jaime

From:

Jenkins, Ray

Sent:

Monday, October 15, 2007 12:03 PM

To:

Bauer, Jaime

Subject: FW: Iluka VPDES renewal

----Original Message----

**From:** Allen, John [mailto:John.Allen@iluka.com] **Sent:** Monday, October 15, 2007 11:52 AM

To: Jenkins, Ray

Subject: Iluka VPDES renewal

Good morning Ray,

I asked about the discharge point for sump 3050. It does indeed run to the Effluent Pond. So Figure 4 in Appendix 1 is correct.

I'm still looking over the data on the landscape pages.

Also, attached is a slightly better copy of the site map I sent you on Friday.

Thanks,

John A. Allen

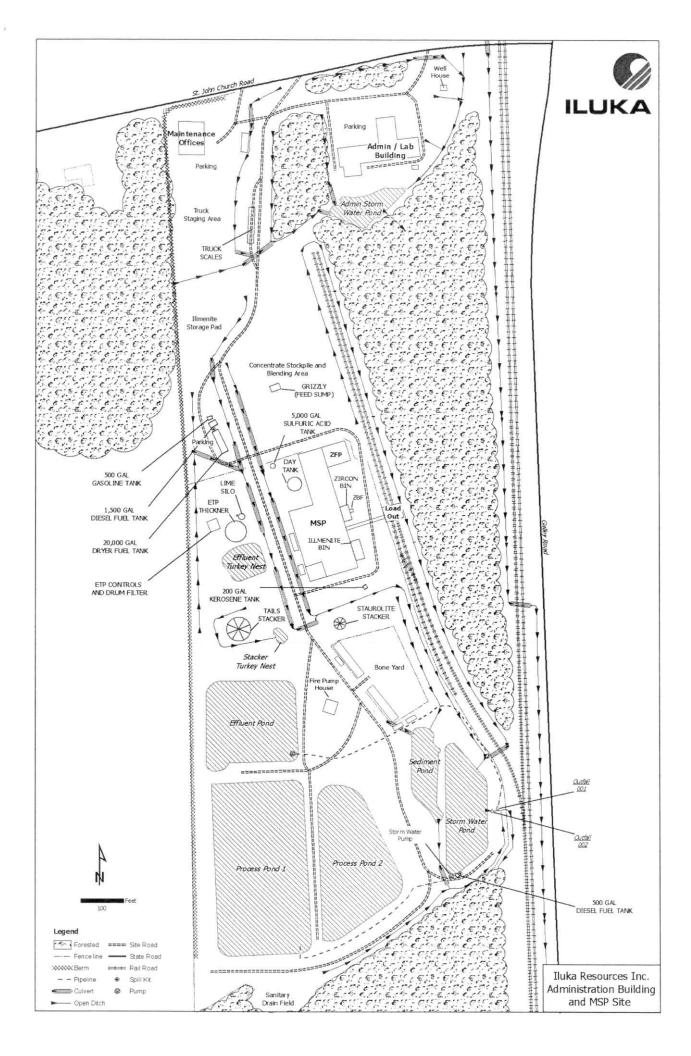
Environmental Officer Iluka Resources Inc

Office: (434)-348-4315 Cell: (804)-943-5611

john.allen@iluka.com <<MSP\_SWPP.pdf>>

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October 29, 2007

Mr. Ray Jenkins DEQ-PRO 4949 A Cox Road Glen Allen, VA 23060

RE: Proposed Changes to VPDES Permits VA0090981 and VAR051217

Mr. Jenkins:

lluka would like to propose the following changes to storm water outfalls from the Mineral Separation Plant (MSP) and Administration / Laboratory Building:

- 1. that the outfall from the Admin Storm Water Pond be removed from Permit # VAR051217 and added to Permit #VA0090981 as outfall 003,
- 2. that the berm currently separating Retention Pond 3 (VA0090981 outfall 002) from the Sediment Pond (VAR051217) be removed or breached, and
- 3. that the Sediment Pond be deepened and its outfall permanently blocked.

These changes would allow all storm water being discharged from the MSP and Administration / Laboratory site to pass through outfalls associated with the site's existing Individual Storm Water Permit #VA0090981, removing the need for the General Permit #VAR051217. A completed Form 2F will be submitted to DEQ for the addition of outfall 003. Please see the enclosed map which shows the proposed modifications to the site.

Please contact me if these proposed changes meet with your approval or if you require additional information.

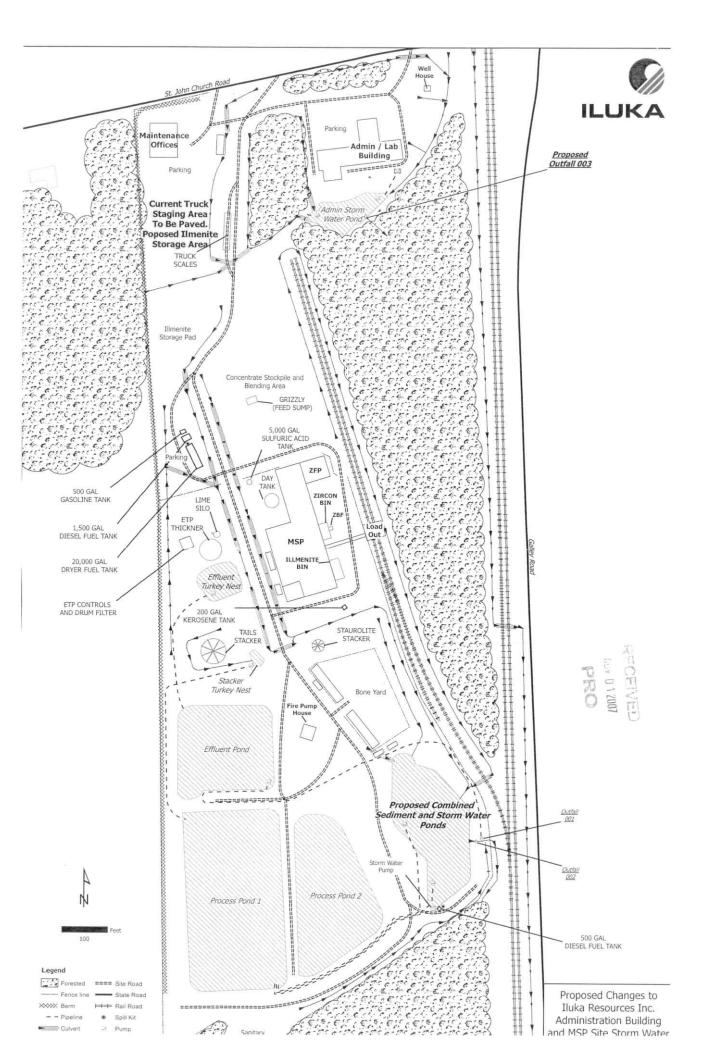
Regards,

John A. Allen

Environmental Officer

Enclosure

Cc: Allan Sale, US Region, General Manager Shawn Conaway, Technical Manager, Virginia Operations Jack Rayburn, EHS&T Supervisor, Virginia Operations





November 16, 2007

Ms. Jaime Bauer Environmental Specialist II DEQ – PRO 4949-A Cox Road Glen Allen, VA 23060

RE: Form 2F Application to Discharge Storm Water Associated With Industrial Activities

Dear Ms. Bauer

Iluka Resources Inc. hereby submits USEPA Form 3510-2F for the Admin Storm Water Pond. This pond and its outfall are currently permitted under the site's General Storm Water Permit VPDES #VAR051217. Iluka would like to remove this pond and its outfall from Permit #VAR051217 and place it under the site's Individual Permit to Discharge Storm Water Associated with Industrial Activities, VPDES Permit #VA0090981, which is currently open for renewal.

Included with this application is an Appendix containing the following documents:

- Figure 1. Location Map Administration/ Laboratory Building and Mineral Separation Plant Site
- Figure 2. Administration/Laboratory Building and MSP Site Layout and Drainage Areas
- Figure 3. Admin Storm Water Pond Inlets, Discharges and Probable Circulation Patterns
- Document 1: Narrative Description of the Site and Processes
- Retention Time Calculation For Admin Storm Water Pond
- Sampling Results
- Pest Control Agreement and MSDSs
- MSDSs for Chemicals used in the Laboratory

If you have any questions or require clarification on any portion of this application package, please do not hesitate to contact me either in my office at 434-348-4315, on my cell phone at 804-943-5611, or via email at john.allen@iluka.com.

Regards,

John A. Allen

Environmental Officer Iluka Resources Inc CEIVED

HOV 1 @ 2007

PRO

EPA ID Number (copy from Item 1 of Form 1) VA0090981

Please print or type in the unshaded areas only.

2F

NPDES

Form Approved. OMB No. 2040-0086 Approval expires 5-31-92

U.S. Environmental Protection Agency Washington, DC 20460

## Application for Permit to Discharge Storm Water Discharges Associated with Industrial Activity

#### Paperwork Reduction Act Notice

Public reporting burden for this application is estimated to average 28.6 hours per application, including time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding the burden estimate, any other aspect of this collection of information, or suggestions for improving this form, including suggestions which may increase or reduce this burden to: Chief, Information Policy Branch, PM-223, U.S. Environmental Protection Agency, 1200 Pennsylvania Avenue, NW, Washington, DC 20460, or Director, Office of Information and Regulatory Affairs, Office of Management and Budget, Washington, DC 20503.

| I. Outfall Location       | Maria was bloom |                  |                 |               |              |            |  |
|---------------------------|-----------------|------------------|-----------------|---------------|--------------|------------|--|
| For each outfall, list th | ne latitude and | I longitude of i | its location to | the nearest 1 | 5 seconds an | d the name | e of the receiving water.                  |
| A. Outfall Number (list)  |                 | B. Latitude      |                 |               | . Longitude  |            | D. Receiving Water (name)                  |
| 003                       | 36.00           | 58.00            | 25.30           | 77.00         | 24.00        | 16.00      | GALLEY SWAMP                               |
|                           |                 |                  |                 |               |              |            | see Fig.1 in the Appendix for location map |
|                           |                 |                  |                 |               |              |            |  |
|                           |                 |                  |                 |               |              |            |  |
|                           |                 |                  |                 |               |              |            |  |
|                           |                 |                  |                 |               |              |            |  |
|                           |                 |                  |                 |               |              |            |  |
|                           |                 |                  |                 |               |              |            |  |
|                           |                 |                  |                 |               |              |            |  |
| No. 10                    |                 |                  |                 |               |              |            |  |
| II. Improvements          |                 |                  |                 |               |              |            |  |

A. Are you now required by any Federal, State, or local authority to meet any implementation schedule for the construction, upgrading or operation of wastewater treatment equipment or practices or any other environmental programs which may affect the discharges described in this application? This includes, but is not limited to, permit conditions, administrative or enforcement orders, enforcement compliance schedule letters, stipulations, court orders, and grant or loan conditions.

| 1. Identification of Conditions, |        | 2. Affected Outfalls |                  |                  | 4.<br>Compli | Final<br>ance Date |
|----------------------------------|--------|----------------------|------------------|------------------|--------------|--------------------|
| Agreements, Etc.                 | number | source of discharge  | 3. Brief Descrip | ption of Project | a. req.      | b. proj            |
| NA                               | NA     | NA                   | NA               |                  | NA           | NA                 |
|                                  |        |                      |                  |                  |              |                    |
|                                  |        |                      |                  |                  |              |                    |
|                                  |        |                      |                  |                  |              |                    |
|                                  |        |                      |                  |                  |              |                    |
|                                  |        |                      |                  |                  | -            |                    |
|                                  |        | 3*                   |                  |                  |              |                    |
|                                  |        |                      |                  |                  |              |                    |
|                                  |        |                      |                  |                  |              |                    |
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|                                  |        |                      |                  |                  |              |                    |
|                                  |        |                      |                  |                  |              |                    |

B: You may attach additional sheets describing any additional water pollution (or other environmental projects which may affect your discharges) you now have under way or which you plan. Indicate whether each program is now under way or planned, and indicate your actual or planned schedules for construction.

#### III. Site Drainage Map

Attach a site map showing topography (or indicating the outline of drainage areas served by the outfalls(s) covered in the application if a topographic map is unavailable) depicting the facility including: each of its intake and discharge structures; the drainage area of each storm water outfall; paved areas and buildings within the drainage area of each storm water outfall, each known past or present areas used for outdoor storage of disposal of significant materials, each existing structural control measure to reduce pollutants in storm water runoff, materials loading and access areas, areas where pesticides, herbicides, soil conditioners and fertilizers are applied; each of its hazardous waste treatment, storage or disposal units (including each area not required to have a RCRA permit which is used for accumulating hazardous waste under 40 CFR 262.34); each well where fluids from the facility are injected underground; springs, and other surface water bodies which received storm water discharges from the facility.

Please see Figure 2 in the Appendix

| 10 173 13VA       | om the Front<br>tive Description of Pollutant  | Sources  | k jan vise j  |  | *           |                                    |
|-------------------|--|--|---|--|-------------|------------------------------------|
| A. For each       |  |  | es (including paved a   | areas and building roofs) drained to the outfall, an   | d an estima | te of the total surface area       |
| Outfall<br>Number | Area of Impervious Surface (provide units)   | Total Area Drained (provide units)   | Outfall<br>Number   | Area of Impervious Surface (provide units)   |             | Total Area Drained (provide units) |
| 003               | ~1.6 ACRES   | 5.2 ACRES  | Number  | (provide urins)  |             | (provide dime)                     |
| to storr          | m water; method of treatment, storage<br>vater runoff; materials loading and acc                                     | e, or disposal; past and pre   | esent materials ma  | years have been treated, stored or dispos<br>anagement practices employed to minimiza<br>equency in which pesticides, herbicides, so   | e contact l | by these materials with            |
| PLEASE RE         | FER TO DOCUMENT 1 in the App   | endix.   |   |  |             |                                    |
| descri            |  | receives, including the sch  |   | ructural control measures to reduce pollution maintenance for control and treatment me   |             |                                    |
| Outfall<br>Number |  |  | List Codes from<br>Table 2F-1                                     |  |             |                                    |
| 003               | (Admin Storm Water Pond) w<br>for suspended solids durin<br>calculation and Figure 3 f<br>submersible pump will be u | e Administration/Lab<br>hich provides approx<br>g the 1 hr/25 year s<br>or a diagram of the<br>sed to mix the pond | rimately 2.5 h<br>storm event (s<br>pond). Grass<br>water to ensu | ing's Storm Water collection por<br>ours of retention and settling t<br>ee Document 2 in the Appendix for<br>lined ditches feed the pond. A<br>re a discharge of relatively<br>e level spreader with weir. | time        | 1-U, 1-O                           |
|                   | ormwater Discharges  |  |   |  |             |                                    |
|                   |  |  |   | ted or evaluated for the presence of nonst<br>Form 2C or From 2E application for the out   |             | discharges, and that al            |
| Au.               | Official Title (type or print)  AN R SALE  RESIDENT  | Signature / / /  | le  |  | Date Sig    | 13/07                              |
| B. Provid         | le a description of the method used, th  | e date of any testing, and t   | he onsite drainage  | points that were directly observed during  | a test.     |                                    |
| lo proces         | s water lines are present wi   | thin the drainage ar   | ea served by  | this outfall. No testing was dee   | emed nec    | essary.                            |
|                   |  |  |   |  |             |                                    |
| /I. Signif        | icant Leaks or Spills  |  | K Balance   |  |             | X                                  |
|                   | existing information regarding the his<br>late date and location of the spill or lea                                 |  |   | r hazardous pollutants at the facility in the<br>sed.  | e last thre | ee years, including the            |
| NA                |  |  |   |  |             |                                    |
|                   |  |  |   |  |             |                                    |

## Continued from Page 2

EPA ID Number (copy from Item 1 of Form 1) VA0090981

| VII. Discharge Information  |  | 5.60年,2015年1月25年  |  |
|---|--|---|--|
|   | ceeding. Complete one set of tables for each outfall. e included on separate sheets numbers VII-1 and VII  |   | pace provided.   |
|   | analysis – is any toxic pollutant listed in table 2F-2 rmediate or final product or byproduct?   | 2F-3, or 2F-4, a substance or a co  | omponent of a substance which you  |
| Yes (list all such pollutants b   | pelow)   | No (go to Section IX)   |  |
| Titanium, Zirconium*, Oil and Gre   | ase.   |   |  |
|   | the zircon sand product produced by the conium. It is believed absent due to the   |   |  |
|   |  |   |  |
|   |  |   |  |
| VIII. Biological Toxicity Testing D   |  |   |  |
| Do you have any knowledge or reason to lead to relation to your discharge within the last 3 | believe that any biological test for acute or chronic to<br>years?   | xicity has been made on any of your   | discharges or on a receiving water in  |
| Yes (list all such pollutants b   | elow)  | No (go to Section IX)   |  |
|   | VII performed by a contract laboratory or consulting and telephone number of, and pollutants   | firm?   |  |
| A. Name   | B. Address   | C. Area Code & Phone No.  | D. Pollutants Analyzed   |
| Primary Laboratories  | 7423 Lee Davis Road<br>Mechanicsville, VA 23111  | 804-559-9004  | See Sampling Results in the Appendix. The results of TCLP extractions from the mineral sand concentrate, ilmenite and zircon products is also provided in the Appendix for your reference. |
| X. Certification  |  | e Conjugation of Constitution Spains and                                      |  |
| that qualified personnel properly gather an<br>directly responsible for gathering the infor | ument and all attachments were prepared under my<br>id evaluate the information submitted. Based on my<br>rmation, the information submitted is, to the best of<br>g false information, including the possibility of fine ar | inquiry of the person or persons who my knowledge and belief, true, according | manage the system or those persons<br>curate, and complete. I am aware that  |
| A. Name & Official Title (Type Or Print)  ALLIAW 17 JALE                                    | PRESIDENT  | B. Area Code and Phone No. 23882  | 348-4300   |
| C. Signature  | la   | D. Date Signed 11/13/0  |  |

## VII. Discharge information (Continued from page 3 of Form 2F)

Part A - You must provide the results of at least one analysis for every pollutant in this table. Complete one table for each outfall. See instructions for additional details.

|  |  |                            |  | Number                     |                                  |                              |
|--|--|----------------------------|--|----------------------------|----------------------------------|------------------------------|
| Pollutant<br>and<br>CAS Number<br>(if available) | Grab Sample<br>Taken During<br>First 20<br>Minutes | Flow-Weighted<br>Composite | Grab Sample<br>Taken During<br>First 20<br>Minutes | Flow-Weighted<br>Composite | of<br>Storm<br>Events<br>Sampled | Sources of Pollutants        |
| Oil and Grease                                   | BQL mg/L   | N/A                        | na   | na                         | 0.00 (1)                         | na                           |
| Biological Oxygen<br>Demand (BOD5)               | 3.6 mg/L   | na                         | na   | na                         | 0.00                             | Unknown                      |
| Chemical Oxygen<br>Demand (COD)                  | 17 mg/L  | na                         | na   | na                         | 0.00                             | Unknown                      |
| Total Suspended<br>Solids (TSS)                  | 30 mg/L  | na                         | 22.1 mg/L  | na                         | 3.00                             | Naturally occuring sediments |
| Total Nitrogen                                   | BQL mg/L   | na                         | na   | na                         | 0.00                             | na                           |
| Total Phosphorus                                 | 0.07 mg/L  | na                         | na   | na                         | 0.00                             | Unknown                      |
| pН   | Minimum 5.50                                       | Maximum 8.12               | Minimum  | Maximum                    | 3.00                             | na                           |

Part B – List each pollutant that is limited in an effluent guideline which the facility is subject to or any pollutant listed in the facility's NPDES permit for its process wastewater (if the facility is operating under an existing NPDES permit). Complete one table for each outfall. See the instructions for additional details and requirements.

|  | (inclu   | ım Values<br>de units)     | Avera<br>(incl                                     | age Values<br>lude units)  | Number                           |                       |
|--|--|----------------------------|--|----------------------------|----------------------------------|-----------------------|
| Pollutant<br>and<br>CAS Number<br>(if available) | Grab Sample<br>Taken During<br>First 20<br>Minutes | Flow-Weighted<br>Composite | Grab Sample<br>Taken During<br>First 20<br>Minutes | Flow-Weighted<br>Composite | of<br>Storm<br>Events<br>Sampled | Sources of Pollutants |
|  |  |                            |  |                            |                                  |                       |
|  |  |                            |  |                            |                                  |                       |
|  |  |                            |  |                            |                                  |                       |
|  |  |                            |  |                            |                                  |                       |
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|  |  |                            | -  |                            |                                  |                       |
|  |  |                            |  |                            |                                  |                       |
|  |  |                            |  |                            |                                  |                       |
|  |  |                            |  |                            |                                  |                       |
|  |  |                            |  |                            |                                  |                       |
|  |  |                            | +  |                            |                                  |                       |
|  |  |                            |  |                            |                                  |                       |
|  |  |                            |  |                            |                                  |                       |
|  |  |                            |  |                            |                                  |                       |
|  |  |                            |  |                            |                                  |                       |
|  |  |                            |  | (6)                        |                                  |                       |
|  |  |                            |  |                            |                                  |                       |
|  |  |                            |  |                            |                                  |                       |
|  |  |                            |  |                            |                                  |                       |
|  |  |                            |  |                            |                                  |                       |
|  |  |                            |  |                            |                                  |                       |
|  |  |                            |  |                            |                                  |                       |
|  |  |                            |  |                            |                                  |                       |

#### Continued from the Front

Part C - List each pollutant shown in Table 2F-2, 2F-3, and 2F-4 that you know or have reason to believe is present. See the instructions for additional details and requirements. Complete one table for each outfall.

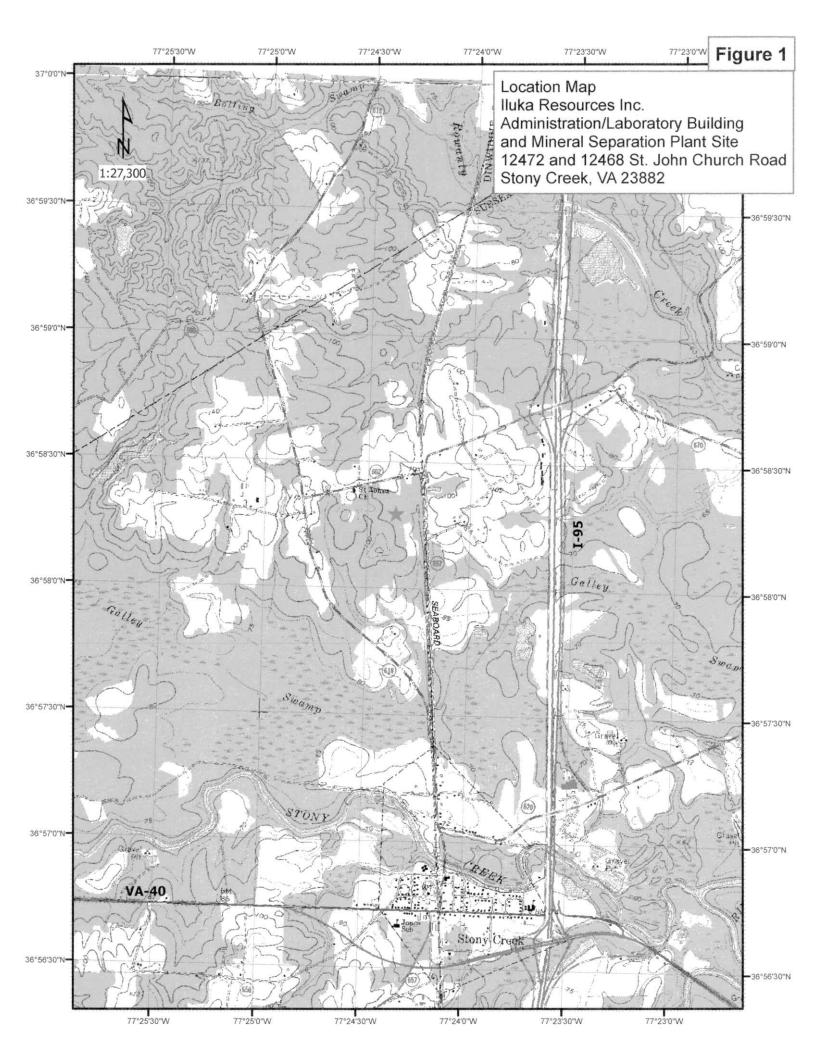
|  | Maximum Values (include units)                     |                            |   | erage Values<br>sclude units) | Number                           |                                      |
|--|--|----------------------------|---|-------------------------------|----------------------------------|--------------------------------------|
| Pollutant<br>and<br>CAS Number<br>(if available) | Grab Sample<br>Taken During<br>First 20<br>Minutes | Flow-Weighted<br>Composite | Grab Sample Taken During First 20 Minutes | Flow-Weighted<br>Composite    | of<br>Storm<br>Events<br>Sampled | Sources of Pollutants                |
| Silver   | BQL mg/L   | na                         | na  | na                            | 3.00                             | na                                   |
| Alkalinity                                       | 86.5 mg/L  | na                         | na  | na                            | 0.00                             | Unknown                              |
| Ammonia-N  | 0.02 mg/L  | na                         | na  | na                            | 0.00                             | Naturally occuring/wildlife          |
| Arsenic  | BQL mg/L   | na                         | na  | na                            | 0.00                             | na                                   |
| Barium   | 0.065 mg/L   | na                         | na  | na                            | 0.00                             | Unknown                              |
| Cadmium  | 0.016 mg/L   | na                         | <0.016 mg/L                               | na                            | 3.00                             | Welding rods/welds                   |
| Chromium   | BQL mg/L   | na                         | na  | na                            | 0.00                             | na                                   |
| Iron   | 13.1 mg/L  | na                         | 4.336 mg/L                                | na                            | 3.00                             | Steel drums/vehicles/natural sources |
| Mercury  | BQL mg/L   | na                         | na  | na                            | 3.00                             | na                                   |
| Potassium  | 6.00 mg/L  | na                         | na  | na                            | 0.00                             | Unknown                              |
| Sodium   | 37.5 mg/L  | na                         | na  | na                            | 0.00                             | Unknown                              |
| Nitrite  | BQL mg/L   | na                         | na  | na                            | 0.00                             | na                                   |
| Nitrate  | 0.04 mg/L  | na                         | na  | na                            | 0.00                             | Unknown                              |
| Lead   | 0.026 mg/L   | na                         | <0.026 mg/L                               | na                            | 3.00                             | Unknown                              |
| Selenium   | 0.008 mg/L   | na                         | <0.008 mg/L                               | na                            | 3.00                             | Unknown                              |
| Sulfate  | 12.3 mg/L  | na                         | na  | na                            | 0.00                             | Natural sources (3)                  |
| TOC  | BQL mg/L   | na                         | na  | na                            | 0.00                             | na                                   |
| Titanium   | BQL mg/L   | na                         | na  | na                            | 0.00                             | na                                   |
| Nickel   | 0.033 mg/L   | na                         | <0.033 mg/L                               | na                            | 3.00                             | Vehicles                             |
| Zinc   | 1.45 mg/L  | na                         | 0.506 mg/L                                | na                            | 3.00                             | Galvinized metal/vehicles            |
|  |  |                            |   |                               |                                  |                                      |
|  |  |                            |   |                               |                                  |                                      |
|  |  |                            |   |                               |                                  |                                      |
|  |  |                            |   |                               |                                  |                                      |
|  |  |                            |   |                               |                                  |                                      |
|  |  |                            |   |                               |                                  |                                      |
|  |  |                            |   |                               |                                  |                                      |
|  |  |                            |   |                               |                                  |                                      |
|  |  |                            |   |                               |                                  |                                      |
|  |  |                            |   |                               |                                  |                                      |

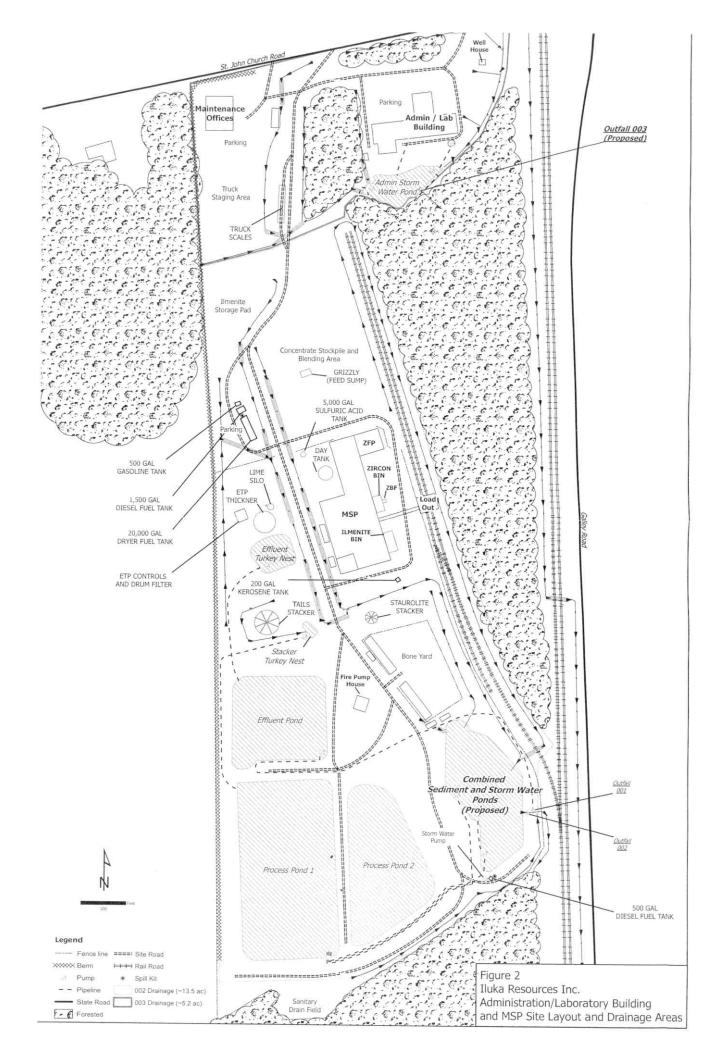
Part D - Provide data for the storm event(s) which resulted in the maximum values for the flow weighted composite sample.

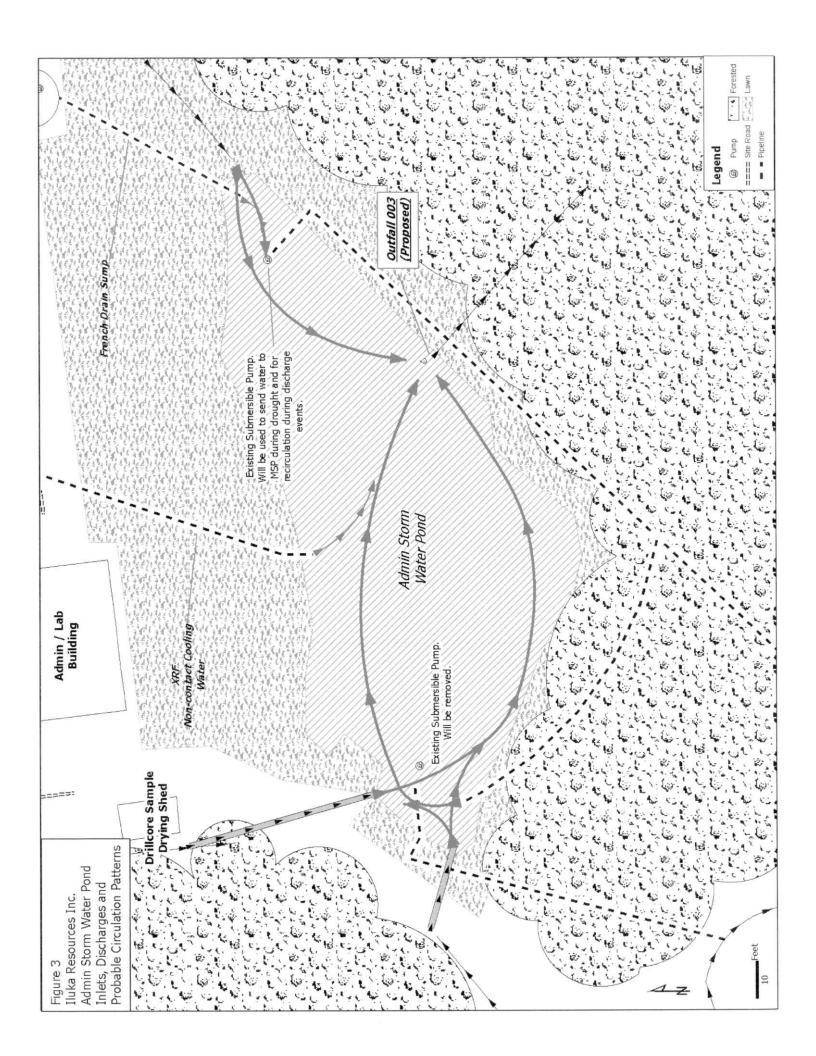
| 1.<br>Date of<br>Storm<br>Event | 2.<br>Duration<br>of Storm Event<br>(in minutes) | 3.<br>Total rainfall<br>during storm event<br>(in inches) | 4. Number of hours between beginning of storm measured and end of previous measurable rain event | 5.  Maximum flow rate during rain event (gallons/minute or specify units) | 6.<br>Total flow from<br>rain event<br>(gallons or specify units) |
|---------------------------------|--|---|--|---|---|
| 7/15/2005                       | unknown  | unknown   | unknown  | unknown   | unknown   |
| 6/3/2005                        | unknown  | unknown   | unknown  | unknown   | unknown   |
| 76/2006                         | unknown  | 4.80  | unknown  | unknown   | unknown   |

<sup>7.</sup> Provide a description of the method of flow measurement or estimate.

A hand held electronic flow meter will be used to obtain the velocity of the flow leaving the outfall. This velocity will be multiplied by the depth of the water passing through the outfall to obtain an estimated volume of discharge.









## ANALYTICAL REPORT

ILUKA RESOURCES INC. 9874 CHEE SAUNDERS 12472 ST. JOHN CHURCH RD. STONY CREEK, VA 23882-0129

Project:

Project Name: VPDES Sampler: CHEE SAUNDERS Lab Number: 00-A114303

Sample ID: MINERAL SAND CONCENTRATE

Sample Type: Solid waste

Site ID:

Date Collected: 8/14/00 Time Collected: 13:45 Date Received: 8/15/00 Time Received: 10:00

#### TCLP Results

| the time of the time of the time of |           |                          |           |              |  |           |                                 |            |           |
|-------------------------------------|-----------|--------------------------|-----------|--------------|--|-----------|---------------------------------|------------|-----------|
|                                     |           |                          | Ħ         | atrix Spike  |  |           |                                 |            |           |
| Analyte                             | Result    | Units                    | Reg Linit | Recovery (X) | Date                                   | Tine      | Analyst                         | Method     | QC Batch  |
|                                     |           | Part 411 701 701 701 701 |           |              | *** *** *** *** *** *** *** *** *** ** | ****      | *** *** *** *** *** *** *** *** |            |           |
|                                     |           |                          |           |              |  | 1277 2092 | 10.100.1010                     | OR WILLIAM | -2122-272 |
| Arsenic                             | (0.100    | Hg/1                     | 5.0       | 106          | 8/22/00                                | 7:52      | 6. Robinson                     | 60108      | 8236      |
| Barium                              | 1.00      | mg/1                     | 100       | 113          | 8/22/00                                | 7:52      | 6. Robinson                     | 6010B      | 8236      |
| Cadmium                             | 0.100     | ng/l                     | 1.0       | 100          | 8/22/00                                | 7:52      | G. Robinson                     | 60108      | 8236      |
| Chronium                            | 0.500     | tig/l                    | 5.0       | 103          | 8/22/00                                | 7:52      | G. Robinson                     | 60108      | 8236      |
| Lead                                | 0.500     | ng/1                     | 5.0       | 98           | 8/22/00                                | 7:52      | G. Robinson                     | 60108      | 8236      |
| Nercury                             | 0.0100    | ng/1                     | 0.20      | 100          | 8/22/00                                | 7:58      | G. McCord                       | 7470A      | 9068      |
| Selenium                            | 0.100     | ng/1                     | 1.0       | 115          | 8/22/00                                | 7:52      | G.Robinson                      | 60108      | 8236      |
| Silver                              | 0.100     | Hg/1                     | 5.0       | 105          | 8/22/00                                | 7:52      | G. Robinson                     | 60108      | 8236      |
| TCLP Extraction                     | Initiated |                          |           |              | 8/16/00                                | 17:30     | K. Bundy                        | 1311       | 6948      |
|                                     |           |                          |           |              |  |           |                                 |            |           |

 $\mathtt{KD}=\mathtt{Mot}$  detected at the report limit. Matrix Spike Recovery for TCLP analytes reported from Natch AC TCLP prepartion follows method 1311, SN-846 Revision 3.

All results reported on a met meight basis.



#### ANALYTICAL REPORT

Laboratory Number: 00-A114303

Sample ID: MINERAL SAND CONCENTRATE

Page 2

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Report Approved By:

Paul E. Lane, Jr., Lab Director Michael H. Dunn, M.S., Technical Director Johnny A. Mitchell, Dir. Technical Serv. Eric S. Smith, Assistant Technical Director Report Date: 8/22/00

Gail A. Lage, Technical Serv. Glenn L. Norton, Technical Serv. Kelly S. Comstock, Technical Serv. Pamela A. Langford, Technical Serv.

Laboratory Certification Number: 00323



### ANALYTICAL REPORT

ILUKA RESOURCES INC. 9874 CHEE SAUNDERS 12472 ST. JOHN CHURCH RD. STONY CREEK, VA 23882-0129

Project:

Project Name: VPDES Sampler: CHEE SAUNDERS Lab Number: 00-A114304 Sample ID: ILMENITE

Sample Type: Solid waste

Site ID:

Date Collected: 8/14/00 Time Collected: 13:45 Date Received: 8/13/00 Time Received: 10:00

#### TCLP Results

|  |                |       | ł         | latrix Spike                               |                                    |       |             |        |          |
|--|----------------|-------|-----------|--|------------------------------------|-------|-------------|--------|----------|
| Analyte  | Result         | Units | Reg Linit | Recovery (%)                               | Date                               | Tine  | Analyst     | Method | AC Batch |
| THE NEW DISCUSSION LINE AND RESERVE THE AND AND LINE AND THE RESERVE THE RESER | PR 100 100 200 |       |           | 100 000 Me 200 200 000 000 000 000 000 000 | 000 DOC 500 DOC 500 DOC 500 FT FIN |       |             |        |          |
| Arsenio  | ⟨ 0.100        | Hg/1  | 5.0       | 106  | 8/22/00                            | 7:52  | 6. Robinson | 6010F  | 8236     |
| Marium   | ₹ 1.00         | 119/1 | 100       | 113  | 8/22/00                            | 7:52  | 6. Robinson | 6010B  | 8236     |
| Cadmium  | ( 0.100        | ng/1  | 1.0       | 100  | 8/22/00                            | 7:52  | 6. Robinson | 6010B  | 8236     |
| Chroniun   | ( 0.500        | Hg/1  | 5.0       | 103  | 8/22/00                            | 7:52  | 6. Robinson | 6010D  | 8236     |
| i_ead  | ( 0.500        | mg/1  | 5.0       | 98   | 8/22/00                            | 7:52  | 6. Robinson | 6010B  | 8236     |
| Mercury  | < 0.0100       | ng/1  | 0.20      | 100  | 8/22/00                            | 7:58  | 6. NoCord   | 7470A  | 9068     |
| Selenium   | ( 0.100        | Hg/1  | 1.0       | 115  | 8/22/00                            | 7:52  | 6. Robinson | 6010B  | 8236     |
| Silver   | ⟨ 0.100        | Hg/1  | 5.0       | 105  | 8/22/00                            | 7:52  | 6. Robinson | 60100  | 8236     |
| TCLP Extraction  | Initiated      |       |           |  | 8/16/00                            | 17:30 | K. Rundy    | 1311   | 6948     |

HD = Not detected at the report limit.

Natrix Spike Recovery for TCLP analytes reported from Batch QC
TCLP prepartion follows method 1311, SW-846 Revision 3.

All results reported on a met meight basis.



#### ANALYTICAL REPORT

Laboratory Number: 00-A114304

Sample ID: ILMENITE

Page 2

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Report Approved By:

Paul E. Lane, Jr., Lab Director Michael H. Dunn, M.S., Technical Director Johnny A. Mitchell, Dir. Technical Serv. Eric S. Smith, Assistant Technical Director Report Date: 8/22/00

Gail A. Lage, Technical Serv. Glenn L. Norton, Technical Serv. Kelly S. Comstock, Technical Serv. Famela A. Langford, Technical Serv.

Laboratory Certification Number: 00323

## Test/America

2960 Foster Creighton Dr Nashville, TN 37204 615-726-0177 Fax: 615-726-0954

### ANALYTICAL REPORT

ILUKA RESOURCES INC. 9874 CHEE SAUNDERS 12472 ST. JOHN CHURCH RD. STONY CREEK, VA 23882-0129

Project:

Project Name: VPDES Sampler: CHEE SAUNDERS Lab Number: 00-A114305

Sample ID: ZIRCON

Sample Type: Solid waste

Site ID:

Date Collected: 8/14/00 Time Collected: 13:45 Date Received: 8/15/00 Time Received: 10:00

TCLP Results

| 2.36.5m3        |           |       | t         | latrix Spike |         |       |             |        |          |
|-----------------|-----------|-------|-----------|--------------|---------|-------|-------------|--------|----------|
| Analyte         | Result    | Units | Reg Linit | Recovery (%) | Date    | Tine  | Analyst     | Method | QC Batch |
|                 |           |       |           |              |         |       |             | ****** |          |
|                 |           |       |           |              |         |       | SEC UP SEE  | 6      | 22222    |
| Arsenic         | ₹ 0.100   | ng/1  | 5.0       | 106          | 8/22/00 | 7:52  | S. Robinson | 60108  | 8236     |
| Barium          | € 1.00    | 11g/1 | 100       | 113          | 8/22/00 | 7:52  | 6. Robinson | 60106  | 8236     |
| Cadniun         | ₹ 0.189   | Hg/1  | 1.0       | 100          | 8/22/00 | 7:52  | G. Robinson | 60108  | 8236     |
| Chronium        | ₹ 0.500   | Hg/1  | 5.0       | 103          | 8/22/00 | 7:52  | G. Robinson | 60108  | 8236     |
| Lead            | ₹ 8.580   | Hg/1  | 5.0       | 78           | 8/22/00 | 7:52  | G. Robinson | 60100  | 8236     |
| Hercury         | < 0.0100  | ng/1  | 0.20      | 100          | 6/22/00 | 7:58  | 6. NoCord   | 7470A  | 9068     |
| Selenium        | ₹ 0.100   | Hg/1  | 1.0       | 115          | 8/22/00 | 7:52  | G. Robinson | 60108  | 8236     |
| Silver          | ₹ 0.100   | ng/1  | 5.0       | 105          | 8/22/00 | 7:52  | G. Robinson | 60108  | 8236     |
| TCLP Extraction | Initiated |       |           |              | 8/16/80 | 17:30 | K. Bundy    | 1311   | 6948     |

ND = Not detected at the report limit.

Matrix Spike Recovery for TCLP analytes reported from Batch RC
TCLP prepartion follows method 1311, SN-846 Revision 3.

All results reported on a met meight basis.



## ANALYTICAL REPORT

Laboratory Number: 00-A114305

Sample ID: ZIRCON

Page 2

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Report Approved By:

Paul E. Lane, Jr., Lab Director Michael H. Dunn, M.S., Technical Director Johnny A. Mitchell, Dir. Technical Serv. Eric S. Smith, Assistant Technical Director Report Date: 8/22/00

Gail A. Lage, Technical Serv. Glenn L. Norton, Technical Serv. Kelly S. Comstock, Technical Serv. Pamela A. Langford, Technical Serv.

Laboratory Certification Number: 00323



Document 1:

NARRATIVE DESCRIPTION OF THE SITE AND PROCESSES

Iluka Resources' Mineral Separation Plant (MSP) and Administration/Laboratory Building is situated on 36.6 acres in Sussex County near the town of Stony Creek (see Figure 1 in the

Appendix).

Storm water drainage for 5.4 acres of the site, including the Administration/Laboratory Building,

Maintenance Office and associated facilities, is provided by Outfall 003 (see Figure 2 in the

Appendix).

1.0 Significant Materials

Significant materials used, proposed for use, or manufactured in the drainage area served by

Outfall 003 include the following items:

• Finished mineral sand products (not currently stored in this drainage area)

Chemicals used in the Laboratory

The Laboratory's chemical supplies are stored either inside the building or in sealed drums

inside of secondary containment devices and so are not exposed to storm water. Iluka plans to

request to store finished mineral sand products within this drainage area if outfall 003 is

approved.

2.0 Methods of Treatment, Storage, and Disposal

2.1 Finished Mineral Sand Products

Iluka plans to seek approval to temporarily store some finished mineral sand product within the

drainage area served by the Admin Storm Water Pond if outfall 003 is approved. This stock pile

would be exposed to storm water. The pollutant of concern associated with storage of the

mineral sand product is the material itself. These minerals are naturally occurring and inert.

None of the RCRA metals were observed in the results of Toxicity Characteristic Leaching

Procedure (TCLP) testing that was performed on mineral sand concentrate and finished product

materials in 2000. The results of this testing can be found in the Sampling Results portion of the

Appendix, along with the analytical results of samples taken from the Admin Storm Water Pond

as required in Form 2F. Additional analytical results from Benchmark monitoring of the Admin



Storm Water Pond is also included in the Appendix. Table 1 lists some of the properties of the minerals processed by the MSP.

Table 1: Properties of Minerals

| Property            | Ilmenite              | Rutile                    | Leucoxene                             | Zircon                        |
|---------------------|-----------------------|---------------------------|---------------------------------------|-------------------------------|
| Chemical Formula    | FeTiO <sub>3</sub>    | TiO <sub>2</sub>          | TiO <sub>2</sub> + FeTiO <sub>3</sub> | ZrSiO <sub>4</sub>            |
| Specific Gravity    | 4.75                  | 4.22                      | 3.95                                  | 4.69                          |
| Magnetic Properties | Conductor<br>Magnetic | Conductor<br>Non-Magnetic | Conductor<br>Weakly Magnetic          | Non-Conductor<br>Non-Magnetic |

As seen in Table 1, the minerals have relatively high SGs ranging from 3.95 (leucoxene) to 4.75 (ilmenite), as compared to Quartz, which has a SG of 2.65. Therefore, it is unlikely that any stockpiled product would be transported by surface water sheet flow resulting from small storm events. Storm water from the mineral product stockpile area will sheet flow until it intersects one of two unlined drainage channels that are located around the east and south sides of the proposed stockpile area. The drainage channels will convey the storm water east to the Admin Storm Water Pond. In the event of a large storm event, minor amounts of mineral sand product could become available for transport to the Admin Storm Water Pond. If the sand reaches the pond, it will settle out and Iluka will reclaim the valuable material from the drainage channels and/or pond. Transport of mineral sands through outfall 003 from the Admin Storm Water pond is highly unlikely.

## 2.2 Chemicals in the Laboratory

The Laboratory's chemical supplies are stored either inside the building or in sealed drums inside of secondary containment devices and so are not exposed to storm water. Waste chemicals from the Lab are stored in sealed drums inside of secondary containment devices and so are not exposed to storm water. These wastes are regularly collected by Safety Kleen for offsite disposal and/or recycling. Spill kits containing a variety of absorbent materials are stationed in the Laboratory. Copies of the MSDSs for all chemicals used in the Lab are included in the Appendix.



## 3.0 Past and Present Management Practices

Iluka has prepared a combined Storm Water Pollution Prevention and SPCC Plan to document procedures used to minimize the contact of materials with storm water runoff. This document is currently being updated. A copy of the SWPP/SPCC Plan is available for inspection in the Environmental, Health and Safety Office at the Administration/Laboratory Building. As mentioned in the above section, chemicals are primarily stored in within the Laboratory or in sealed drums within secondary containment devices. When this is not possible, the chemicals are stored in sealed containers and placed on paved surfaces in designated areas.

## 4.0 Pesticide and Herbicide Use

Herbicides are not generally used at the site. A monthly service is used to control insects and rodents in the MSP and Administration/Laboratory Building. A copy of the pest control agreement and MSDS sheets for the chemicals used on-site are included in the Appendix.

## RETENTION TIME CALCULATION FOR ADMIN STORM WATER POND

## Flow from the 1 hour / 25 year storm event was calculated using the Rational Method:

$$Q = C \times i \times A$$

Q = Flow (cfs)

C = Runoff Coefficient

i = Inches of rain produced by the 1 hour / 25 year storm event

A = Acres of drainage area

As described in the Virginia Department of Conservation and Recreation's *Virginia Erosion and Sediment Control Handbook*, Third Edition, 1992.

## The following values were used in the calculation:

The runoff coefficient (C) for the site was estimated by weighted average as shown below.

| Land Use             | Area (acres) |   | Runoff<br>Coefficient<br>(C) |   | Weighted C |
|----------------------|--------------|---|------------------------------|---|------------|
| Flat, clay soil lawn | 0.2          | X | 0.15                         | = | 0.03       |
| Sandy, flat lawn     | 0.5          | X | 0.05                         | = | 0.025      |
| Asphalt              | 1.2          | X | 0.70                         | = | 0.84       |
| Gravel lot           | 1.7          | X | 0.40                         | = | 0.68       |
| Metal Roof           | 0.4          | X | 1.00                         | = | 0.40       |
| Woodland             | 1.0          | Χ | 0.10                         | = | 0.10       |
| Totals               | 5.0          |   |                              |   | 2.08       |

Based on this, the weighted average runoff coefficient for the site is calculated to be:  $C = \frac{2.08}{5.0} = 0.42$ 

Rainfall expected from the 1 hour / 25 year storm event (i) is approximately **2.75** inches. This is based on the average of the City of Richmond and Greensville County curve values obtained from the DCR ESC Handbook.

The area (A) served by the Admin Storm Water Pond is approximately **5.2 acres** (including the pond itself).

Therefore:

Q = 
$$0.42 \times 2.75$$
 in  $\times 5.2$  ac  
=  $6.0$  cubic feet per second  
 $\Rightarrow 2,700$  gallons per minute

The volume of Retention Pond B is approximately **0.4 million gallons**.

Therefore, retention time for runoff produced by the 1 hour / 25 year storm is:

SAMPLING RESULTS

## Primary Laboratories, Inc.

Results

14-Jun-05

Date Sampled: Work Order No:

03-Jun-05 0506030-02

Client ID:

Admin. Stormwater Pond (1), (2)

| Cheff ID. Admin. Stormwater Fond (1), (2) |        |           |                      |               |                                  |          |  |  |
|---|--------|-----------|----------------------|---------------|----------------------------------|----------|--|--|
| Test                                      | Final  | Reporting | Units of             | EPA Test      | Date                             | Tech.    |  |  |
| Description                               | Result | Limit     | Measure              | Method        | Analyzed                         | Initials |  |  |
| TSS                                       | 13.0   | 1.0       | mg/L                 | 160.2         | 10-Jun-05<br>at 9:30             | MS       |  |  |
| рН  | 6.57   | -         | pH Units             | 150.1         | 10-Jun-05                        | MS       |  |  |
| Turbidity                                 | 10.5   | -         | NTU                  | Nephelometric | at 11:00<br>6-Jun-05<br>at 14:10 | HV       |  |  |
| Hardness                                  | 50.0   | 1.0       | mg/L                 | 130.2         | 6-Jun-05                         | NA       |  |  |
|   |        |           | as CaCO <sub>3</sub> |               | at 8:30                          |          |  |  |
|   |        |           |                      |               |                                  |          |  |  |

## Primary Laboratories, Inc. Results

14-Jun-05

Date Sampled:

03-Jun-05

Work Order No:

0506030-02 Admin Stormwater Pond (1) (2)

| WORK Order 140. | 61K C14C1 140. 6500050-02 |                |          |             |                       |          |  |  |  |
|-----------------|---------------------------|----------------|----------|-------------|-----------------------|----------|--|--|--|
| Client ID:      | Admin. Storn              | nwater Pond (1 | ), (2)   |             |                       |          |  |  |  |
| Test            | Final                     | Reporting      | Units of | EPA Test    | Date                  | Tech.    |  |  |  |
| Description     | Result                    | Limit          | Measure  | Method      | Analyzed              | Initials |  |  |  |
| Total Metals    |                           |                |          |             |                       |          |  |  |  |
| Antimony        | <0.100                    | 0.100          | mg/L     | 200.2/200.7 | 10-Jun-05             | HV       |  |  |  |
|                 |                           |                |          |             | at 10:58              |          |  |  |  |
| Arsenic         | 0.014                     | 0.005          | mg/L     | 200.2/206.2 | 9-Jun-05              | HV       |  |  |  |
| D III           | 10.010                    | 0.040          |          | 200 2/200 7 | at 15:30              | 1.15.7   |  |  |  |
| Beryllium       | <0.010                    | 0.010          | mg/L     | 200.2/200.7 | 10-Jun-05<br>at 10:58 | HV       |  |  |  |
| Cadmium         | 0.016                     | 0.010          | mg/L     | 200.2/200.7 | 9-Jun-05              | HV       |  |  |  |
| Guarrian        | 0.010                     | 0.010          | 1119/2   | 200.2/200.7 | at 13:56              |          |  |  |  |
| Copper          | 0.367                     | 0.020          | mg/L     | 200.2/200.7 | 10-Jun-05             | HV       |  |  |  |
|                 |                           |                |          |             | at 10:58              |          |  |  |  |
| Iron            | 13.1                      | 0.050          | mg/L     | 200.2/200.7 | 10-Jun-05             | HV       |  |  |  |
| Leval           | 0.000                     | 0.005          | /1       | 200 2/220 2 | at 10:58              | 1.07     |  |  |  |
| Lead            | 0.026                     | 0.005          | mg/L     | 200.2/239.2 | 7-Jun-05<br>at 15:00  | HV       |  |  |  |
| Manganese       | 0.294                     | 0.020          | mg/L     | 200.2/220.7 | 10-Jun-05             | HV       |  |  |  |
| Manganese       | 0.204                     | 0.020          | l mg/L   | 200.2/220.7 | at 10:58              |          |  |  |  |
| Mercury         | <0.0002                   | 0.0002         | mg/L     | 245.1       | 7-Jun-05              | HV       |  |  |  |
|                 |                           |                |          |             | at 14:25              |          |  |  |  |
| Nickel          | 0.033                     | 0.020          | mg/L     | 200.2/200.7 | 9-Jun-05              | HV       |  |  |  |
| 0.1.            |                           | 0.005          |          | 000 0/070 0 | at 13:56              | 1.07     |  |  |  |
| Selenium        | <0.005                    | 0.005          | mg/L     | 200.2/270.2 | 6-Jun-05<br>at 12:41  | HV       |  |  |  |
| Silver          | <0.001                    | 0.001          | mg/L     | 200.2/272.2 | 9-Jun-05              | HV       |  |  |  |
| 5.1.7 51        | 0.001                     | 0.001          | 19. =    |             | at 12:00              |          |  |  |  |
| Zinc            | 1.45                      | 0.010          | mg/L     | 200.2/200.7 | 10-Jun-05             | HV       |  |  |  |
|                 |                           |                |          |             | at 10:58              |          |  |  |  |

Signature:

Parry L. Baragg√

Laboratory Manager

These analytical results are based upon materials provided by the client and are intended for the exclusive use of the client. These analytical results represent the best judgement of Primary Laboratories, Inc. Primary Laboratories, Inc. assumes no responsibility, express or implied, as to the interpretation of the analytical results contained in this report. This report is not to be reproduced except with the written approval of Primary Laboratories, Inc.

## Primary Laboratories, Inc.

7423 Lee Davis Road • Mechanicsville, VA 23111 • Telephone (804) 559-9004 • Fax (804) 559-9306



## ANALYTICAL LABORATORY REPORT

26-Jul-05

ILUKA Resource, Inc. Attn: Kevin Rideout 12472 St. John Church Stony Creek, Virginia 23882

Date Received:

15-Jul-05

Date Sampled:

15-Jul-05

Work Order No:

0507097-01

Client ID:

| Client ID:          | Admin. Stom     | iwater#1 Gra       | D                |                    |                  |                   |
|---------------------|-----------------|--------------------|------------------|--------------------|------------------|-------------------|
| Test<br>Description | Final<br>Result | Reporting<br>Limit | Units of Measure | EPA Test<br>Method | Date<br>Analyzed | Tech.<br>Initials |
|                     |                 |                    |                  |                    |                  |                   |
| TSS                 | 27.6            | 1.0                | mg/L             | 160.2              | 22-Jul-05        | MS                |
|                     |                 |                    |                  |                    | at 10:00         |                   |
| рН                  | 6.89            | <b>*</b> 0         | pH Units         | 150.1              | 22-Jul-05        | MS                |
|                     |                 |                    |                  |                    | at 11:17         |                   |
| Turbidity           | <1.0            | 1.0                | NTU              | Nehelometric       | 19-Jul-05        | HV                |
|                     |                 |                    |                  |                    |                  |                   |

pH Temperature = 20.6 Degrees C.

## Primary Laboratories, Inc. Results

26-Jul-05

Date Sampled:

15-Jul-05

Work Order No:

0507097-02

| Client ID: | Admin. Stormwater | # | 2 |
|------------|-------------------|---|---|
|            |                   |   |   |

| Client ID:                      | Admin. Storr | water # 2 |                              |             |                       |            |
|---------------------------------|--------------|-----------|------------------------------|-------------|-----------------------|------------|
| Test                            | Final        | Reporting | Units of                     | EPA Test    | Date                  | Tech.      |
| Description                     | Result       | Limit     | Measure                      | Method      | Analyzed              | Initials   |
| Hardness                        | 27.1         | 0.1       | mg/L<br>as CaCO <sub>3</sub> | 130.2       | 21-Jul-05<br>at 8:30  | NA         |
| Metals                          |              |           |                              |             |                       |            |
| Antimony                        | <0.100       | 0.100     | mg/L                         | 200.2/200.7 | 18-Jul-05             | HV         |
| Arsenic                         | <0.005       | 0.005     | mg/L                         | 200.2/206.2 | at 13:32<br>20-Jul-05 | HV         |
|                                 |              |           |                              |             | at 14:45              |            |
| Beryllium                       | <0.010       | 0.010     | mg/L                         | 200.2/200.7 | 18-Jul-05             | HV         |
|                                 |              |           |                              |             | at 13:32              |            |
| Cadmium                         | <0.010       | 0.010     | mg/L                         | 200.2/200.7 | 18-Jul-05             | HV         |
| 0                               | 0.029        | 0.020     | ma/l                         | 200.2/200.7 | at 13:32<br>18-Jul-05 | HV         |
| Copper                          | 0.028        | 0.020     | mg/L                         | 200.2/200.7 | at 13:32              | TIV        |
| Iron                            | 1.26         | 0.050     | mg/L                         | 200.2/200.7 | 18-Jul-05             | HV         |
|                                 |              |           |                              |             | at 13:32              |            |
| Lead                            | < 0.005      | 0.005     | mg/L                         | 200.2/239.2 | 19-Jul-05             | HV         |
|                                 |              |           |                              |             | at 14:15              | 501.401150 |
| Manganese                       | 0.082        | 0.020     | mg/L                         | 200.2/200.7 | 18-Jul-05             | HV         |
|                                 | .0.0000      | 0.0000    |                              | 245.4       | at 13:32<br>20-Jul-05 | HV         |
| Mercury                         | <0.0002      | 0.0002    | mg/L                         | 245.1       | at 15:35              | HV         |
| Nickel                          | <0.020       | 0.020     | mg/L                         | 200.2/200.7 | 18-Jul-05             | HV         |
| MICKEL                          | 40.020       | 0.020     | ling/L                       | 200.2/200.7 | at 13:32              |            |
| Selenium                        | < 0.005      | 0,005     | mg/L                         | 200.2/270.2 | 20-Jul-05             | HV         |
| Control Control Control Control |              |           |                              |             | at 15:25              |            |
| Silver                          | <0.001       | 0.001     | mg/L                         | 200.2/272.2 | 21-Jul-05             | HV         |
|                                 |              |           |                              |             | at 9:45               |            |
| Zinc                            | 0.021        | 0.010     | mg/L                         | 200.2/200.7 | 18-Jul-05             | HV         |
| 1 (A) American Annual           |              |           |                              |             | at 13:32              |            |

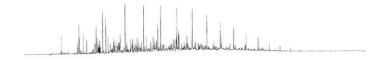
Laboratory Manager

Date: 7/24/05

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## Primary Laboratories, Inc.

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## ANALYTICAL LABORATORY REPORT

21-Jul-06

ILUKA Resource, Inc. Attn: Kevin Rideout 12472 St. John Church Stony Creek, Virginia 23882

Date Received:

07-Jul-06

Date Sampled:

06-Jul-06

Work Order No:

0607026-01

| Client ID:  | Admin Storr | n #1      |          |                     |   |          |
|-------------|-------------|-----------|----------|---------------------|---|----------|
| Test        | Final       | Reporting | Units of | EPA Test            | Date  | Tech.    |
| Description | Result      | Limit     | Measure  | Method              | Analyzed                                    | Initials |
| TSS         | 30          | 1         | mg/L     | 160.2               | 11-Jul-06                                   | MS       |
| рН          | 5.50        |           | pH Units | 150.1               | at 13:30<br>12-Jul-06                       | MS       |
| Temp for pH | 8.4         | -         | Degree C |                     | 12-Jul-06<br>at 10:05                       | MS       |
| Turbidity   | 7.8         | -         | NTU      | Nephelometric/180.1 | 1,500 10 10 10 10 10 10 10 10 10 10 10 10 1 | AB       |

Date Sampled:

06-Jul-06

Work Order No:

0607026-03

Client ID:

Concord Storm Water Outfall S001 #1

| Client ID: Concord Storm Water Outlan Soot #1 |        |           |          |                     |                       |          |  |
|---|--------|-----------|----------|---------------------|-----------------------|----------|--|
| Test  | Final  | Reporting | Units of | EPA Test            | Date                  | Tech.    |  |
| Description                                   | Result | Limit     | Measure  | Method              | Analyzed              | Initials |  |
| TSS   | 89.2   | 1.0       | mg/L     | 160.2               | 11-Jul-06<br>at 13:30 | MS       |  |
| рН  | 5.67   | -         | pH Units | 150.1               | 12-Jul-06             | MS       |  |
| Temp for pH                                   | 9.2    | -         | Degree C |                     | 12-Jul-06             | MS       |  |
| rempter pri                                   |        |           |          |                     | at 10:10              |          |  |
| Turbidity                                     | 41.5   | _         | NTU      | Nephelometric/180.1 | 14-Jul-06             | AB       |  |
| , and and                                     |        |           |          |                     | at 14:05              |          |  |

## Primary Laboratories, Inc. Results

21-Jul-06

Date Sampled:

06-Jul-06

Work Order No:

0607026-02

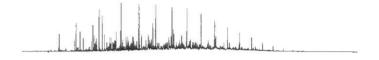
Client ID:

Admin Storm #2

| Client ID:         | Admin Storn | 1 #2      |                              |             | ,                                 |          |
|--------------------|-------------|-----------|------------------------------|-------------|-----------------------------------|----------|
| Test               | Final       | Reporting | Units of                     | EPA Test    | Date                              | Tech.    |
| Description        | Result      | Limit     | Measure                      | Method      | Analyzed                          | Initials |
| Hardness           | 21.1        | 0.1       | mg/L<br>as CaCO <sub>3</sub> | 130.2       | 14-Jul-06<br>at 14:00             | NA       |
| Metals<br>Antimony | <0.100      | 0.100     | mg/L                         | 200.2/200.7 | 17-Jul-06<br>at 11:50             | АВ       |
| Arsenic            | <0.005      | 0.005     | mg/L                         | 200.2/206.2 | 20-Jul-06                         | AB       |
| Beryllium          | <0.010      | 0.010     | mg/L                         | 200.2/200.7 | at 11:54<br>17-Jul-06<br>at 11:50 | AB       |
| Cadmium            | <0.010      | 0.010     | mg/L                         | 200.2/200.7 | 17-Jul-06                         | AB       |
| Copper             | 0.066       | 0.020     | mg/L                         | 200.2/200.7 | at 11:50<br>17-Jul-06<br>at 11:50 | АВ       |
| Iron               | 2.07        | 0.050     | mg/L                         | 200.2/200.7 | 17-Jul-06<br>at 11:50             | AB       |
| Lead               | <0.005      | 0.005     | mg/L                         | 200.2/239.2 | 17-Jul-06<br>at 11:50             | AB       |
| Manganese          | 0.061       | 0.020     | mg/L                         | 200.2/200.7 | 17-Jul-06<br>at 11:50             | AB       |
| Mercury            | <0.0002     | 0.0002    | mg/L                         | 245.1       | 14-Jul-06<br>at 14:48             | AB       |
| Nickel             | <0.020      | 0.020     | mg/L                         | 200.2/200.7 | 17-Jul-06<br>at 11:50             | AB       |
| Selenium           | 0.008       | 0.005     | mg/L                         | 200.2/270.2 | 19-Jul-06                         | AB       |
| Silver             | <0.001      | 0.001     | mg/L                         | 200.2/272.2 | at 14:06<br>17-Jul-06             | AB       |
| Zinc               | 0.047       | 0.010     | mg/L                         | 200.2/200.7 | at 15:07<br>17-Jul-06<br>at 11:50 | AB       |

## Primary Laboratories, Inc.

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## ANALYTICAL LABORATORY REPORT

31-Oct-07

Iluka Resources, Inc Attn: Kevin Rideout

12472 St Johns Church Road Stoney Creek, VA 23882

Project:

MSP Outfall 003

Date Received:

23-Oct-07

Date Sampled:

23-Oct-07

Work Order No:

0710198-01

Client ID:

| Client ID:       | Outfall 003 |           |                              |                                    | 500000000000000000000000000000000000000 |          |          |
|------------------|-------------|-----------|------------------------------|------------------------------------|---|----------|----------|
| Test             | Final       | Reporting | Units of                     | Standard                           | Date                                    | Time     | Tech.    |
| Description      | Result      | Limit     | Measure                      | Methods (18)                       | Analyzed                                | Analyzed | Initials |
| O&G              | <0.9        | 0.9       | mg/L                         | 5520 B                             | 30-Oct-07                               | 9:30     | AS       |
| BOD              | 3.6         | 2.0       | mg/L                         | 5210 B                             | 24-Oct-07                               | 7:45     | NA       |
| COD              | 17.0        | 2.0       | mg/L                         | 5220C                              | 30-Oct-07                               | 12:00    | NA       |
| TSS              | 17.8        | 1.0       | mg/L                         | 2540 D                             | 29-Oct-07                               | 8:30     | AS       |
| TKN              | <1.0        | 1.0       | mg/L                         | 4500 N/NH <sub>3</sub>             | 24-Oct-07                               | 15:00    | NA       |
| рН               | 8.12        | -         | pH Units                     | 4500H <sup>†</sup> B               | 31-Oct-07                               | 11:30    | HV       |
| Total Phosphorus | 0.07        | 0.01      | mg/L                         | 4500P E                            | 28-Oct-07                               | 10:00    | NA       |
| Alkalinity       | 86.5        | 0.1       | mg/L<br>as CaCO <sub>3</sub> | 2320 B                             | 24-Oct-07                               | 15:30    | NA       |
| Ammonia          | 0.02        | 0.01      | mg/L                         | 4500NH <sub>3</sub> F              | 24-Oct-07                               | 8:00     | NA       |
| тос              | <1.00       | 1.00      | mg/L                         | 5310 C                             | 29-Oct-07                               | 7:30     | РВ       |
| Nitrate          | 0.04        | 0.01      | mg/L                         | 4500 NO <sub>3</sub> -E            | 26-Oct-07                               | 13:00    | NA       |
| Nitrite          | <0.005      | 0.005     | mg/L                         | 4500 NO <sub>2</sub> B             | 25-Oct-07                               | 12:00    | NA       |
| Sulfate          | 12.3        | 2.0       | mg/L                         | 4500SO <sub>4</sub> <sup>2</sup> E | 29-Oct-07                               | 14:00    | NA       |

## Primary Laboratories, Inc. Results

31-Oct-07

Project:

MSP Outfall 003

Date Received:

23-Oct-07

Date Sampled:

23-Oct-07

Work Order No:

0710198-01

Client ID:

Outfall 003

| Offerit ID. | Outian 003 |           |          |              |           |          |          |
|-------------|------------|-----------|----------|--------------|-----------|----------|----------|
| Test        | Final      | Reporting | Units of | Standard     | Date      | Time     | Tech.    |
| Description | Result     | Limit     | Measure  | Methods (18) | Analyzed  | Analyzed | Initials |
| Metals      |            |           |          |              |           |          |          |
| Arsenic     | <0.050     | 0.050     | mg/L     | 3030E/3120B  | 29-Oct-07 | 14:02    | HV       |
| Barium      | 0.065      | 0.020     | mg/L     | 3030E/3120B  | 29-Oct-07 | 14:02    | HV       |
| Chromium    | <0.020     | 0.020     | mg/L     | 3030E/3120B  | 29-Oct-07 | 14:02    | HV       |
| Iron        | 0.926      | 0.050     | mg/L     | 3030E/3120B  | 29-Oct-07 | 14:02    | HV       |
| Lead        | <0.050     | 0.050     | mg/L     | 3030E/3120B  | 29-Oct-07 | 14:02    | HV       |
| Mercury     | <0.0002    | 0.0002    | mg/L     | 3112 B       | 25-Oct-07 | 12:52    | HV       |
| Potassium   | 6.00       | 1.00      | mg/L     | 3030E/3120B  | 29-Oct-07 | 14:02    | HV       |
| Selenium    | <0.050     | 0.050     | mg/L     | 3030E/3120B  | 29-Oct-07 | 14:02    | HV       |
| Silver      | <0.020     | 0.02      | mg/L     | 3030E/3120B  | 29-Oct-07 | 14:02    | HV       |
| Sodium      | 37.5       | 5.0       | mg/L     | 3030E/3120B  | 29-Oct-07 | 14:02    | HV       |
| Titanium    | <0.020     | 0.020     | mg/L     | 3030E/3120B  | 29-Oct-07 | 14:02    | HV       |

Note: Samples received in laboratory on ice.

Parry L. Bradg Laboratory Manager

These analytical results are based upon materials provided by the client and are intended for the exclusive use of the client. These analytical results represent the best judgement of Primary Laboratories, Inc. Primary Laboratories, Inc. assumes no responsibility. express or implied, as to the interpretation of the analytical results contained in this report. This report is not to be reproduced except with the written approval of Primary Laboratories, Inc.

# PEST CONTROL AGREEMENT AND MSDS SHEETS

PHINTED IN U.S.A. H

## Pest Control Service Agreement

## HOUCHINS PEST CONTROL, INC.

2775 County Drive PETERSBURG, VA 23803 (804) 732-2930 Fax (804) 732-6842

1077

| CUSIONLI  |  | SERVICE LOCATION        |   |  |  |  |  |
|---|--|-------------------------|---|--|--|--|--|
| Iluka Resources, Inc.   |  | 1                       |   | 2000   |  |  |  |
| 12472 St. Johns Church 1  | Road   |                         |   |  |  |  |  |
| Stops Ctools 17A 22992  |  | PERSON TO DE            | CONTACTION  | SERVICE PHONE  |  |  |  |
| Stony Creek, VA 23882   | YPE OF PROPERTY TO BIL SERVICE D   |                         | de adole, a proprie   |  |  |  |  |
| 434-246-8016  |  |                         |   |  |  |  |  |
| DATE SERVICE REGINS   | XPIRATION DATE   | RENEWAL                 | SERVICE TO BE PERFORMED                                     |  |  |  |  |
| PESTS TO BE CONTROLLED:   |  | 1                       | MONTHLY DOUA  | RTERLY   OTHER   |  |  |  |
| Roaches, ants, spiders a  | and rodents  |                         | THE RESIDENCE IN  |  |  |  |  |
|   |  |                         |   |  |  |  |  |
|   |  |                         | n in commence and the commence                              |  |  |  |  |
|   |  |                         |   |  |  |  |  |
| SPECIAL INSTRUCTIONS:   |  | 0 10 100                |   |  |  |  |  |
| Treat hallways, restroom  | s, breakrooms and offi   | ces; sp                 | ray outside perim   | eter of blue   |  |  |  |
| building and both traile  | rs. Use bait and glue  | boards                  | for control of ro   | dents  |  |  |  |
|   |  |                         | TOT COMMITTED OF TO   | delics.  |  |  |  |
| PERSON SEE A COMPANIE   |  | 2 174 111               |   |  |  |  |  |
| Termites and clovermites  | are not included in t  | L.J                     | tt  |  |  |  |  |
| TERMS AND CONDITIONS:   | in beholder in all   | nis con                 | tract.  | 4  |  |  |  |
|   |  |                         |   |  |  |  |  |
|   |  |                         |   |  |  |  |  |
|   |  |                         |   |  |  |  |  |
|   |  |                         |   |  |  |  |  |
|   |  |                         |   |  |  |  |  |
| SERVICE GUARANTEE: We agree Service Agreement. All labor and federal, state and city regulations. | e to apply chemicals to control abo<br>materials will be furnished to provi    | ve-named<br>de the mos  | posts in accordance with te<br>t efficient pest control and | rms and conditions of this<br>maximum safety required by |  |  |  |
| SERVICE RENEWAL: This agreem this agreement by giving thirty day                                  | nent shall be for an Initial period of<br>s written notice before any expirati | one year, a<br>on date. | and will renew itself annuall                               | y unless either party cancels                            |  |  |  |
|   |  |                         | 14  | 1 (1   |  |  |  |
| ANNUAL<br>AGREEMENT CHARGE  | \$   | BY                      | 91/21   | 1  |  |  |  |
| INITIAL SERVICE CHARGE  | \$   | COMPA                   | (AUTHORIZED SIGNATURE)                                      | 10/2/06  |  |  |  |
| MONTHLY/QUARTERIES  | \$ 150.00  | FOR                     |   |  |  |  |  |
| ***************************************   | \$   | CUSTO                   | MEH(AUTHORIZED SKINATUIR!)                                  | DATE   |  |  |  |

## MSDS

CONTRAC®

Date of Issue: FEBRUARY 1995

| 3699 KINSMAN BLVD., M  | NC.<br>IADISON   | WI 53704   | VJD CAR  | (608) 2  | 41-0202   |   | t your local<br>Control Center   |
|--|--|--|--|--|---|---|--|
| PRODUCT NAME:<br>CONTRAC®  |  |  |  |  |   | CAS NO.:  | 6.7  |
| CHEMICAL FAMILY<br>Coumarin  |  |  | 3-13-(4'-Br  | NAME & SYNO:<br>omo-[1,1'-biph<br>y-2H-1-benz  | cnvll-4-vl)-3   | -hvdroxv-1-   | phenylpropyll-   |
| CHEMICAL FORMULA:  |  |  | 4-hydrox   | y-2H-1-benz  | opyran-2-c  | one   | · · · · · · · · · · · · · · · · · · ·  |
| C30H23BrO4   |  |  | 1  | Bromadiolor  | 1e  |   |  |
| S  | ECTIO  | ON I. HA   | ZARĐO  | US INGRI   | EDIENTS   | S   |  |
| ACTIVE INGREDIENTS:<br>Bromadiolone  |  |  |  |  |   | 0.005   | 500 mg/kg<br>based on<br>beagle dog  |
| SECTIO   | N. II  | DIIVELCA   | I DATA   | OF ACTI  | VE INC  | DEDIEN  | Late.  |
| APPEARANCE & ODOR:   |  | TLAR WEIGHT  |  | OF ACTI  |   |   | C GRAVITY:   |
| White odorless powder<br>VAPOR DENSITY (AR=1):   | COLOR  | 527.4  | В  | 200-2<br>ULK DENSITY:  | 10°C  | BOILIN  | NA<br>g point:   |
| NA<br>Vapor pressure:  | SOLUBI   | White  |  | 0.33 g   |   | 200.00000   | NA<br>PATION RATE:   |
| NA NA  | 12 n   | ng/l (water)   |  | NA NA  | * 2.  | EVAPOR  | NA<br>NA   |
| SECTIO   | )N III.  | FIRE &   | EXPLOS   | SION DAT   | A OF P  | RODUC   | T  |
| FLASII POINT F (METHOD USED):<br>NA  |  |  | ELAN   | MABLE LIMIT:<br>NA   |   | AUI   | OIGNITION TEMP:<br>NA  |
| EXTINGUISHING MEDIA:   | 14   | R MOLENCIA   | Marie es   | 1.76%  |   |   | .5565  |
| Extinguish w<br>SPECIAL FIRE FIGHTING PRO-   |  |  | iert gas.  |  |   |   |  |
| None<br>UNUSUAL FIRE OR EXPLOSION  | MAZARD   | 95:  |  |  |   |   |  |
| None   |  |  |  |  |   |   |  |
| SECTION IV. 1  |  |  |  | DATA OF  | ACTIV   | E INGRI   | EDIENT   |
| STABILITY:<br>Stable   | COND   | NA<br>NA   | OID:   |  |   |   |  |
| POLYMERIZATION:  | COND   | TIONS TO AV  | OID:   |  |   |   |  |
| Will not occur. INCOMPATABILITY (MATERIA)  | LS TO AVO  | NA<br>MDs:   | 1 "  | AZARDOUS DE  |   |   |  |
| Strong Bases   | 0  | ECTION   | V TOY  | Carbon Mor   | March 1977  | arbon Dio   | cide   |
| LD50,ORAL (INGESTION)  |  | LD50.DERMAI  | 100000000000000000000000000000000000000  | ACT): 11% AT   | 1   | TON: 1,C56  | (Ps. At)   |
| LD50, ORAL (INGESTION)<br>1 125 mg/kg [Rat]<br>1.75 mg/kg [Mous  | e]   |  | kg [Rabbit]  |  |   | mg/1 x 1 h  |  |
| FISH LC50:<br>1.4 mg/l [Rambow Tr  |  | Non-irr  |  | 0N: 11° - A  |   | M.:<br>g/kg [Dog  | (TECH)   |
| 5.0 mg/l [Biuegiii su  | miisn)   | 13.011-111   |  |  |   |   |  |
|  |  |  | namg   |  | 10 10   | Sub Inch  |  |
| CECTIO   | N: N/I   | HE . L TH  |  | D DITI   |   |   |  |
| RIMARY ROUTE OF ENTRY-   |  | Turnious o   | HAZAR  | D DATA   | OF PRO  | DUCT  |  |
| RIMARY ROUTE OF ENTRY:<br>x Ingestion Skin & Ey  | e contact  | Turnious o   | HAZAR  | RD DATA<br>ENERALLY AGG  | OF PRO  | DUCT  | SIGNAL WORD:<br>Caution  |
| PRIMARY ROUTE OF ENTRY:  X Ingestion Skin & Fy Inhalation Skin absorbed HAZARDS:   | ee contact<br>orption  | MEDICAL C  | HAZAR ONDITIONS G  | ENERALLY AGG<br>None<br>DMS OF EXPOSE  | OF PRO  | DUCT<br>EXPOSURE:   | SIGNAL WORD:<br>Caution  |
| PRIMARY ROUTE OF ENTRY:  X Ingestion Skin & Fy Inhalation Skin absorbated HAZARDS:  1- Caution: May be irrit EMERGENCY FIRST AID PROCEE  | ating.   | MEDICAL C  | HAZAR ONDITIONS G  | ENERALLY AGG<br>None<br>DMS OF EXPOSE<br>clotting abilit   | OF PRO  | DDUCT<br>EXPOSURE:  | SIGNAL WORD:<br>Caution  |
| PRIMARY BOUTE OF ENTRY: Ingestion Skin & FxInhalation Skin absorbed.  BEALTH HAZARDS:  1- Caution: May be irrit  | ating.   | MEDICAL C  | HAZAR ONDITIONS G  2 NS & SYMPTO lay reduce  | ENERALLY AGG<br>None<br>DMS OF EXPOSE<br>clotting abilit<br>up & water.  | OF PRO  | DDUCT<br>EXPOSURE:  | SICNAL WORD-<br>Caution  |
| PRIMARY ROLTE OF ENTRY:  \[ \times \text{ Ingestion } \times \text{ Skin & Fy} \]  Inhalation \( -\text{Skin abst} \)  Inhalation \( -\text{Skin abst} \)  In Caution: May be irrit  EMERGENCY FIRST AID PROCEE  Eyes: Flush with plenty of  Ingestion: Administer V  overdoses. R   | ating<br>of water.<br>I water at the control of the contr | Skin: Wa   | HAZAR ONDITIONS G  NS & SYMPTO lay reduce ush with sociscularly of   | ENERALLY AGG<br>None<br>OMS OF EXPOSE<br>clotting abilit<br>ap & water.<br>r orally as inc   | OF PRO  | DDUCT<br>EXPOSURE:<br>lood and co   | SICNAL WORD-<br>Caution  |
| PRIMARY ROUTE OF ENTRY:  \[ \lambda \text{ [Skin & Fy   Inhalation } \] \[ \lambda \text{ [Skin alise]} \] \[ \lambda \text{ [Laution: May be irrite } \] \[ \lambda \text{ [Caution: May be irrite } \] \[ \left \text{ [Caution: May be irrite } \] \[ \left \text{ [First all PROCEE } \] \[ \text{ [Syes: Flush with plenty of } \] \[ \lambda \text{ [May be irrite } \] \[ \text{ [May be irrite } \]  | ating<br>of water.<br>I water at the control of the contr | Skin: Wa   | HAZAR ONDITIONS G  NS & SYMPTO lay reduce ush with sociscularly of   | ENERALLY AGG<br>None<br>OMS OF EXPOSE<br>clotting abilit<br>ap & water.<br>r orally as inc   | OF PRO  | DDUCT<br>EXPOSURE:<br>lood and co   | SICNAL WORD-<br>Caution  |
| PRIMARY BOUTE OF ENTAY.  ** Ingestion — Skin & F.  ** Inhabition — Skin also  BEALTHHAZARDS:  1- Caution: May be irrit  EMBERGENCY FIRST AID PROCEE  EYES: Flush with plenty of  Ingestion: Administer V  Overdoses. R  SPECIAL PROTECTION INSO  | ating<br>of water.<br>I water at the control of the contr | Skin: Wa   | HAZAR ONDITIONS G  NS & SYMPTO lay reduce ush with sociscularly of   | ENERALLY AGG<br>None<br>OMS OF EXPOSE<br>clotting abilit<br>ap & water.<br>r orally as inc   | OF PRO  | DDUCT<br>EXPOSURE:<br>lood and co   | SICNAL WORD-<br>Caution  |
| PRIMARY BOUTE OF ENTAY.  ** Ingestion — Skin & F.  ** Inhabition — Skin also  BEALTHHAZARDS:  1- Caution: May be irrit  EMBERGENCY FIRST AID PROCEE  EYES: Flush with plenty of  Ingestion: Administer V  Overdoses. R  SPECIAL PROTECTION INSO  | ating.  ating.  tress f water.  Fitamin I epeat as   | Skin: Wa<br>Skin: Wa<br>K i intrarmu<br>necessary b  | HAZAR ONDITIONS G  2 NS 4 SYMPT day reduce ush with sociscularly or passed on m  | ENERALLY AGG<br>None<br>OMS OF EXPOSE<br>clotting abilit<br>up & water,<br>orally as inconitoring of   | OF PRO  | DDUCT<br>EXPONERE:<br>lood and co<br>on: None,<br>bishydroxy<br>oin times.  | sicval words<br>Caution<br>ause bleeding.  |
| PRIMARY BOLTE OF ESTRY 2. Ingestion — Skin & F. 2. Ingestion — Skin abst [EMATH HAZARIS: 1. Caution: May be irrit EMERGEACY FIRST AID PROFEE Eyes: Flush with plenty of Ingestion: Administer V overdoses. R SPECIAL PROTECTION INFO None  SECTION VII. C RESPIRATOR TAPE:   | ating.  ating.  tress f water.  Fitamin I epeat as   | Skin: Wa<br>Skin: Wa<br>K i intrarmu<br>necessary b  | HAZAR ONDITIONS G  2 NS 4 SYMPT day reduce ush with sociscularly or passed on m  | ENERALLY AGG<br>None<br>OMS OF EXPOSE<br>clotting abilit<br>up & water,<br>orally as inconitoring of   | OF PRO  | DDUCT<br>EXPONERE:<br>lood and co<br>on: None,<br>bishydroxy<br>oin times.  | sicval words<br>Caution<br>ause bleeding.  |
| PRIMARY BOLTE OF ENTRY  Langestion — Skin & F.  Inhabilation — Skin altos  LEALTH HAZARDS:  L-Caution: May be irrit  EMERGERCY Flush with plenty of  Ingestion: Administer V  overdoses. R  SPECIAL PROTECTION INFO  None  SECTION VII. C  RESPIRATOR TYPE:  None  | ating.  ating.  tress f water.  Fitamin I epeat as   | Skin: Was Skin: Was necessary b  | HAZAR ONDITIONS G  SS 4 SYMPT day reduce ush with souscularly or assed on m  | ENERALLY AGG<br>None<br>OMS OF EXPOSE<br>clotting abilit<br>up & water,<br>orally as inconitoring of   | OF PRC REVATED BY REJ y of the bl Inhalatic dicated in 1 prothromb  | DDUCT<br>EXPONERE:<br>lood and co<br>on: None,<br>bishydroxy<br>oin times.  | sicval words<br>Caution<br>ause bleeding.  |
| PRIMARY BOUTE OF ENTRY  L Ingestion — Skin & F.  Inhabition — Skin also  Inhabition — Skin also  Inhabition: May be irrit  EMERGENCY FIRST AID PROCEE  EYES, Flush with plenty of  Ingestion: Administer V  Overdoses. R  SPECIAL PROTECTION INFO  None  SECTION VII. C  RESPIRATOR TYPE:  None  EYE PROTECTION:  None  OTHER PROTECTIVE MEASURE  OTHER PROTECTIVE MEASURE  OTHER PROTECTIVE MEASURE  OTHER PROTECTIVE MEASURE   | ating ating of water. Fitamin I epeat as semantion   | Skin: Was Skin: Was necessary b  | HAZAR ONDITIONS G  2 NS 4 SYMPT flay reduce ush with sociscularly or passed on m   | ENERALLY AGG<br>None<br>OMS OF EXPOSE<br>clotting abilit<br>up & water,<br>orally as inconitoring of   | OF PRO  | DDUCT<br>EXPONERE:<br>lood and co<br>on: None,<br>bishydroxy<br>oin times.  | sacyat words<br>Caution<br>ause bleeding.  |
| PRIMARY BOLTE OF ESTRY  Langestion — Skin & F.  Ingstall — Skin abs  BEALTH HAZARIS  1 - Caution: May be irrit  EMERCENCY FIRST AID PROCEE  EYES: Flish with plenty of  Ingestion: Administer V  overdoses. R  SPECIAL PROTECTION INFO  NONE  SECTION VII. C  RESPRATOR TYPE:  None  NONE  | ating ating of water. Fitamin I epeat as semantion   | Skin: Was Skin: Was necessary b  | HAZAR ONDITIONS G  SS 4 SYMPT day reduce ush with souscularly or assed on m  | ENERALLY AGG<br>None<br>OMS OF EXPOSE<br>clotting abilit<br>up & water,<br>orally as inconitoring of   | OF PRC REVATED BY REJ y of the bl Inhalatic dicated in 1 prothromb  | DDUCT<br>EXPONERE:<br>lood and co<br>on: None,<br>bishydroxy<br>oin times.  | sacyat words<br>Caution<br>ause bleeding.  |
| PRIMARY ROLTE OF ESTRY  Langestion — Skin & F.  Inhabilition — Skin abst  Inhabilition — May be irrite  Eyes: Flush with plenty of  Ingestion: Administer V  overdoses. R  SPECIAL PROTECTION INFO  None  SECTION VII. C  RESPIRATOR TAPE:  None  OTHER PROTECTIVE MEASURY  None  SECTION  SECTION  SECTION  SECTION  SECTION  | ating. ot res f water. //itamin l epeat as semartion   | Skin: We K + intrarmun necessary b   | HAZAR ONDITIONS G  ? NS & SYMPTI lay reduce ush with souscularly or sased on m  ROTECT DOET gloves  R LEAK   | ENERALLY AGG<br>None<br>OMS OF EXPOSE<br>clotting abilit<br>up & water,<br>orally as inconitoring of   | OF PRO RAVATED BY  RE: y of the bl  Inhalatic dicated in h prothromb  SURES (  VENTILATIC None  | DDUCT EXPONERE: lood and cr on: None. bishydroxy bin times.  OF PRO   | sicxal words<br>Caution<br>ause bleeding.  |
| PRIMARY ROLTE OF ESTRY  Langestion — Skin & F.  Inglation — Skin abs  EMERICANS  1 - Caution: May be irrit  EMERICANY FIRST AID PROCEE  EYES: Flush with plenty of  Ingestion: Administer V  overdoses. R  SPECIAL PROTECTION INFO  None  SECTION VII. C  RESPIRATOR TYPE:  None  EYE PROTECTIVE MEASURE  NONE  SECTION  SECTI | ating. ot res f water. //itamin l epeat as semartion   | Skin: We K + intrarmun necessary b   | HAZAR ONDITIONS G  ? NS & SYMPTI lay reduce ush with souscularly or sased on m  ROTECT DOET gloves  R LEAK   | ENERALLY AGG None MS OF EXPOSIC Clothing abilit up & water. orally as inconitoring of  | OF PRO RAVATED BY  RE: y of the bl  Inhalatic dicated in h prothromb  SURES (  VENTILATIC None  | DDUCT EXPONERE: lood and cr on: None. bishydroxy bin times.  OF PRO   | sicxal words<br>Caution<br>ause bleeding.  |
| PRIMARY ROLTE OF ESTRY  Langestion — Skin & F.  Inhabilition — Skin abst  Inhabilition — May be irrite  Eyes: Flush with plenty of  Ingestion: Administer V  overdoses. R  SPECIAL PROTECTION INFO  None  SECTION VII. C  RESPIRATOR TAPE:  None  OTHER PROTECTIVE MEASURY  None  SECTION  SECTION  SECTION  SECTION  SECTION  | ating. ot res f water. //itamin l epeat as semartion   | Skin: We K + intrarmun necessary b   | HAZAR ONDITIONS G  ? NS & SYMPTI lay reduce ush with souscularly or sased on m  ROTECT DOET gloves  R LEAK   | ENERALLY AGG None MS OF EXPOSIC Clothing abilit up & water. orally as inconitoring of  | OF PRO RAVATED BY  RE: y of the bl  Inhalatic dicated in h prothromb  SURES (  VENTILATIC None  | DDUCT EXPONERE: lood and cr on: None. bishydroxy bin times.  OF PRO   | sicxal words<br>Caution<br>ause bleeding.  |
| PRIMARY ROLTE OF ESTRY  Langestion — Skin & F.  Inghalation — Skin & F.  Inhalation — Skin after  Eyes: Flush with plenty of  Ingestion: Administer V  overdoses. R  SPECIAL PROTECTION INFO  None  SECTION VII. C  RESPIRATOR TYPE:  None  SECTION  STEPS TO BE TAKEN IN CASE M  General clean-up.  WASTE BISPOSAL METHOD:   | ee contacts ating. ating. target water further water furth       | Skin; Wa Skin; Wa Ki i intrarrius necessary b  | HAZAR ONDITIONS G  \$ 5  | ENERALLY AGG VOICE | OF PRO RAVATED BY  BE2 y of the bl  Inhalatic dicated in I prothromb  SURES  VENTILATIC NONE  URE OF  | DDUCT EXPOSERE: lood and co on: None. bishydroxy oin times.  OF PRO   | shexal words Caution ause bleeding.  |
| PRIMARY ROLTE OF ESTRY  Langestion — Skin & F.  Inghalation — Skin after  Inghalation — May be irrite  Eyes: Flush with plenty of  Ingestion: Administer V  overdoses. R  SPECIAL PROTECTION INFO  None  SECTION VII. C  RESPIRATOR TYPE  None  OTHER PROTECTIVE MEASUR  NONE  SECTION  STEPS TO BE TAKEN IN CASE M  General clean-up.   | or contact        | Skin; Wa Skin; Wa Ki i intrarrius necessary b  | HAZAR ONDITIONS G  \$ 5  | ENERALLY AGG VOICE | OF PRO RAVATED BY  BE2 y of the bl  Inhalatic dicated in I prothromb  SURES  VENTILATIC NONE  URE OF  | DDUCT EXPOSERE: lood and co on: None. bishydroxy oin times.  OF PRO   | shexal words Caution ause bleeding.  |
| PRIMARY BOLTE OF ESTRY  Langestion — Skin & F.  Inghalation — Skin & F.  Inhalation — Skin abs  III — Caution: May be irrit  EMERICANY FIRST AID PROCEE  EYES: Flush with plenty of  Ingestion: Administer V  overdoses. R  SPECIAL PROTECTION INFO  NONE  SECTION VII. C  RESPIRATOR TYPE:  NONE  SECTION  WASTE DISPOSAL METHOD:  Product that cannot be use  Product that cannot be use   | or contactor or co       | Skin: Wa Skin: Wa K   intrarrate necessary b  CLOVES Rubit  SPILL OI IS RELEASED O   | HAZAR ONDITIONS G  SYMPTI  SYM | ENERALLY AGG VOICE | OF PRO BRAVATED BY BRE BY of the bl Inhalatic dicated in 1 prothromb  SURES VENILLATE None  URE OF  | DDUCT EXPOSE RE- LOOK AND TO THE PROPERTY OF PROPERTY | sicval words Caution ause bleeding.  |
| PRIMARY BOLTE OF ENTAY  Langestion — Skin &  | re contactor of the contact of the c       | Skin; Waxing Skin; | HAZAR ONDITIONS OF THE PROPERTY OF THE PROPERT | ENERALLY AGG VOICE | OF PRO RAVATED BY RE: y of the bl Inhalatic dicated in 1 prothromb  SURES  VENTILATE None  URE OF   | DDUCT EXPOSURE: lood and ci on: None. bishydroxy oin times.  PRODU  PRODU  Caroling to  | sicxal words. Caution ause bleeding. Vecoumarin  DUCT  applicable Feder  RODUCT                  |
| PRIMARY BOLTE OF ESTRY  Langestion — Skin & Fast  Inhalation — Skin & Skin abs  ENALTH HAZARIS  1 - Caution: May be irrit  ENERGENCY FIRST AID PROCEE  EYEST Flush with plenty of  Ingestion: Administer V  overdoses. R  SPECIAL PROTECTION INFO  None  SECTION VII. C  RESPIRATOR TYPE:  None  SECTION  SECTION  SECTION  SECTION  SECTION  SECTION  WASTE DISPOSAL METHOD:  Product that cannot be use  State or Local procedure:  SECTION IX. SPI  | ee contactory atting.  HARSA  HARSA  VIII.  VIII.  ECCONTR  VIII.  ECCIAL  ECCIAL  ECCIAL  ITERIOR OF THE PROPERTY OF THE PROP       | Skin: Wa Skin: Wa Ki intrarrium necessary b Science Spill Oil Spill Spil | HAZAR ONDITIONS OF THE PROPERTY OF THE PROPERT | ENERALLY AGG VOICE | OF PRO RAVATED BY RE: y of the bl Inhalatic dicated in 1 prothromb  SURES  VENTILATE None  URE OF   | DDUCT EXPOSURE: lood and ci on: None. bishydroxy oin times.  PRODU  PRODU  Caroling to  | ssexat words Caution ause bleeding.  Vecoumarin  DUCT  applicable Feder  RODUCT                  |
| PRIMARY BOLTE OF ENTRY  Langestion — Skin & F.  Inghalation — Skin abs  ELATH HAZARIS:  I - Caution: May be irrit EMERGENCY FIRST AIP PROFEE  Eyes: Flush with plenty of  Ingestion: Administer V overdoses. R SPECIAL PROTECTION INFO  None  SECTION VII. C RESPIRATOR TYPE: None  SECTION  X. SPINITION  SECTION  | re contactor of the con       | Skin: Wa Ski | HAZAR ONDITIONS OF THE PROPERTY OF THE PROPERT | ENERALLY AGG VOICE | OF PRO RAVATED BY RE: y of the bl Inhalatic dicated in 1 prothromb  SURES  VENTILATE None  URE OF   | DDUCT EXPOSURE: lood and ci on: None. bishydroxy oin times.  PRODU  PRODU  Caroling to  | ssexat words Caution ause bleeding.  Vecoumarin  DUCT  applicable Feder  RODUCT                  |
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BAYER CROP SCIENCE

P.O. Box 4913 Hawthorn Road Kansas City, MO 64120-0013

TRANSPORTATION EMERGENCY NON-TRANSPORTATION CALL CHEMTREC: 800-424-9300 BAYER EMERGENCY PHONE...: (800) 414-0244 INTERNATIONAL: 703-527-3887 BAYER INFORMATION PHONE.: (800) 842-8020 CHEMICAL PRODUCT IDENTIFICATION: PRODUCT NAME..... TEMPO SC Ultra Premise Spray PRODUCT CODE..... 21648 CHEMICAL FAMILY....: Pyrethroid Insecticide CHEMICAL NAME.....: Cyano (4-fluoro-3-phenoxyphenyl) methyl 3-(2,2 -dichloroethenyl)-2,2-dimethylcyclopropanecarboxylate SYNONYMS..... beta-cyfluthrin FORMULA..... C22 H18 C12 F N O3 COMPOSITION/INFORMATION ON INGREDIENTS: INGREDIENT NAME /CAS NUMBER EXPOSURE LIMITS CONCENTRATION (%) \*\*\*\*\* HAZARDOUS INGREDIENTS FCR 4545 Technical (beta-cyfluthrin)

Specific chemical identity is withheld as a trade secret.

OSHA: Not Established ACGIH: Not Established

ACGIH: Not Established

68359-37-5 OSHA: Not Established

1-3 %

11.8 %

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HAZARDS IDENTIFICATION: \*\*\*\*\*\*\*\*\*\*\*\* EMERGENCY OVERVIEW \* CAUTION! Color: Beige; Form: Liquid; Off-white to beige \* viscous liquid suspension; Odor: Chalky; Harmful if \* inhaled; Harmful if absorbed through skin; Causes eye \* irritation. \*\*\*\*\*\*\*\*\* POTENTIAL HEALTH EFFECTS: ROUTE(S) OF ENTRY...... Inhalation; Skin Contact; Skin Absorption; Eye Contact HUMAN EFFECTS AND SYMPTOMS OF OVEREXPOSURE: ACUTE EFFECTS OF EXPOSURE....: Skin and mucous membrane irritation may occur from contact with the product and produce symptoms such as itching, stinging, skin reddening or rash. Paresthesia (a tingling or burning sensation on the surface of the skin) may also result from skin contact. These are frequently reported symptoms associated with sufficient dermal exposure to alpha-cyano (Type II) synthetic pyrethroids and normally subside without treatment within 24 hours. The onset of these symptoms usually occurs 2-12 hours after exposure. The effects are temporary and are reversible. Based on the EPA Toxicity Category criteria, this material is mildly toxic by the oral and dermal routes of exposure. In addition, animal studies have shown that it can cause mild irritation to the

conjunctiva of the eye with all irritation resolving within 7 days.

CHRONIC EFFECTS OF EXPOSURE...: Based on animal studies, no adverse effects or symptoms would be expected from chronic exposure to this material.

CARCINOGENICITY..... : This product is not listed by NTP, IARC or regulated as a carcinogen by OSHA.

#### MEDICAL CONDITIONS

AGGRAVATED BY EXPOSURE.....: No specific medical conditions are known which may be aggravated by exposure to this product. As with all materials which can cause upper respiratory tract irritation, persons with a history of asthma, emphysema, or hyperreactive airways disease may be more susceptible to overexposure.

#### 4. FIRST AID MEASURES:

FIRST AID FOR EYES.....: Hold eyelids open and flush with copious amounts of water for 15 minutes. Call a physician if irritation develops or persists

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MSDS Rage 2 Continued on next page

#### 4. FIRST AID MEASURES (Continued)

after flushing.

FIRST AID FOR SKIN....: Remove contaminated clothing immediately. Wash skin with soap and water, preferably preceded by a waterless hand cleaner. Get medical attention if irritation develops or persists. If signs of intoxication (poisoning) occur, get medical attention immediately.

FIRST AID FOR INHALATION: If a person is overcome by excessive exposures to aerosols or vapors of this material, remove to fresh air or uncontaminated area. If not breathing, give artificial respiration, preferably mouth-to-mouth. Get medical attention as soon as possible.

FIRST AID FOR INGESTION.: If ingestion is suspected, call a physician or poison control center. Drink one or two glasses of water and induce vomiting by touching back of throat with finger, or, if available, by administering syrup of ipecac. If syrup of ipecac is available, administer 1 tablespoonful (15 mL) of syrup of ipecac followed by 1 to 2 glasses of water. If vomiting does not occur within 20 minutes, repeat the dose once. Do not induce vomiting or give anything by mouth to an unconscious person.

NOTE TO PHYSICIAN.....: ANTIDOTE: No specific antidote is available. Treat victim symptomatically. Published data indicate vitamin E acetate can prevent and/or mitigate symptoms of paresthesia caused by synthetic pyrethroids. In case of poisoning, it is also requested that Bayer Corp., Agriculture Division, Kansas City, Missouri, be notified. Telephone: 1-800-414-0244

#### 5. FIRE FIGHTING MEASURES:

FLASH POINT..... Greater than 200 F (93 C) EXTINGUISHING MEDIA..... Water; Foam; Dry Chemical

SPECIAL FIRE FIGHTING PROCEDURES: Keep out of smoke. Cool exposed containers with water spray. Fight fire from upwind position. Use self-contained breathing equipment. Contain runoff to prevent entry into sewers or waterways. Equipment or materials involved in pesticide fires may become contaminated.

#### 6. ACCIDENTAL RELEASE MEASURES:

SPILL OR LEAK PROCEDURES.....: Isolate area and keep unauthorized people away. Do not walk through spilled material. Avoid breathing vapors and skin contact. Remove sources of ignition if combustible or flammable vapors may be present and ventilate area. Wear proper protective equipment. Dike contaminated area with absorbent granules, soil, sand, etc. If large spill, material should be recovered. Small spills can be absorbed with absorbent granules, spill control pads, or any absorbent material. Carefully sweep up absorbed spilled material. Place in covered container for reuse or disposal. Scrub contaminated area with soap and water. Use dry absorbent materials such as clay granules to absorb and

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# 6. ACCIDENTAL RELEASE MEASURES (Continued)

collect wash solution for proper disposal. Contaminated soil may have to be removed and disposed. Do not allow material to enter streams, sewers, or other waterways or contact vegetation.

## HANDLING AND STORAGE: STORAGE TEMPERATURE(MIN/MAX): 0 F/30 day avg. not to exceed 100 F SHELF LIFE..... : Time/temperature-dependent : Contact Bayer for specific information. SPECIAL SENSITIVITY...... Not cstablished HANDLING/STORAGE PRECAUTIONS: Store in a cool, dry area designated specifically for pesticides. PERSONAL PROTECTION: EYE PROTECTION REQUIREMENTS..... Goggles should be used to prevent liquid from getting into eyes. SKIN PROTECTION REQUIREMENTS...... Avoid skin contact. Wear long sleeves and HAND PROTECTION REQUIREMENTS.....: Chemical-resistant gloves such as neoprene VENTILATION REQUIREMENTS..... : Control exposure levels through the use of general and local exhaust ventilation. RESPIRATOR REQUIREMENTS...... When respiratory protection is necessary under the conditions of use, wear a NIOSH-approved organic vapor respirator with dust and mist filter. ADDITIONAL PROTECTIVE MEASURES....: Clean water and soap should be available for washing in case of eye or skin contamination. Waterless hand cleaner use is often more effective than soap and water. Sensitive areas of the skin and mucous membranes can become contaminated indirectly. Educate and train employees in safe use of the product. Follow all label instructions. Launder clothing separately after use. Wash thoroughly after handling. PHYSICAL AND CHEMICAL PROPERTIES: PHYSICAL FORM..... Liquid APPEARANCE..... Off-white to beige viscous liquid suspension COLOR.... Beige ODOR..... Chalky MOLECULAR WEIGHT..... 434.3 (for beta-cyfluthrin) pH ..... 7-8

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BOILING POINT..... Not established MELTING/FREEZING POINT...: Approx. 20 F

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#### 9. PHYSICAL AND CHEMICAL PROPERTIES (Continued)

VISCOSITY.....: 1060 cps @ 23 C

SOLUBILITY IN WATER ...: Not established

SPECIFIC GRAVITY ...: 1.06 @ 20 C/20 C

BULK DENSITY...: Not established

VAPOR PRESSURE ...: 7.2 x 10-9 mm Hg @ 20 C (for beta-cyfluthrin)

#### 10. STABILITY AND REACTIVITY:

STABILITY..... This is a stable material.

HAZARDOUS POLYMERIZATION ...: Will not occur.

INCOMPATIBILITIES..... Alkaline media; reacts with methanol; incompatible

with many disinfectants.

INSTABILITY CONDITIONS....: Not established DECOMPOSITION PRODUCTS....: Not established

#### 11. TOXICOLOGICAL INFORMATION:

Only acute studies have been performed on this product as formulated. The non-acute information pertains to the active ingredient, cyfluthrin technical, and its enriched isomer mixture, BAY FCR 4545 technical.

#### ACUTE TOXICITY

ORAL LD50.....: Male rat: 960 mg/kg -- Female rat: 1150 mg/kg

DERMAL LD50.....: Male and Female Rat: >2000 mg/kg

INHALATION LC50...: 4 hr exposure to Liquid Aerosol: Male and Female Rat: >1.72 mg/L (analtyical) -- 1 hr exposure to Liquid Aerosol (extrapolated from 4 hr): Male and Female Rat: >6.88 mg/L (analytical)

EYE EFFECTS.....: Rabbit: Mild irritation to the conjunctiva was observed with all irritation cleared within 7 days post-treatment.

SKIN EFFECTS.....: Rabbit: Not a dermal irritant

SENSITIZATION....: Guinea pig: Not a dermal sensitizer

SUBCHRONIC TOXICITY...: FCR 4545: In a 13 week dog study, FCR 4545 was administered at dictary concentrations of 10, 60 or 360 ppm. Effects included vomiting and diarrhea after feeding, decreased body weight gain, and motor disturbances in the hind limbs. The no-observed effect-level (NOEL) was 60 ppm. In a 13 week study using rats, FCR 4545 was administered at dietary concentrations of 30, 125 or 500 ppm. Effects included reduced body weight gains and feed consumption, uncoordinated gait, and skin injuries of the neck and head from excessive preening due to the local irritant effect of the test material. The NOEL was 125 ppm. In a 4 week inhalation study, rats were exposed to FCR 4545 at liquid aerosol concentrations of 0.2, 2.7 or 23.5 mg/m3. Effects observed included ungroomed fur, piloerection, hyper- and hypoactivity, reduced body weight gains, reduced organ weights (thymus and spleen), and hematological changes. The NOEL was 0.2 mg/m3 based on decreased body weight gains. CYFLUTHRIN: In a 3 week dermal toxicity study, cyfluthrin

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#### 11. TOXICOLOGICAL INFORMATION (Continued)

was administered to rats for 6 hours/day at dose levels of 100, 340 or 1000 mg/kg. Animals received a total of 17-18 applications in a period of 22-23 days. An additional control and high-dose group were treated and maintained for 14-15 days following treatment so as to ascertain the extent of recovery. Effects observed included reduced feed consumption, red nasal discharge, urine stains, and findings at the dose site (scabbing, crusty, discolored and raised zones). Histologically, epidermal and dermal alterations in treated skin were observed in animals of the mid- and high-dose groups. Similar, but slightly less severe microscopic alterations were also observed in the high-dose recovery group. The overall NOEL was 100 mg/kg. In a 13 week inhalation study, rats were exposed to cyfluthrin at acrosol concentrations of 0.09, 0.71 or 4.51 mg/m3 for 6 hours/day, 5 days/week. The NOEL was 0.09 mg/m3 based on reduced body weight gains.

CHRONIC TOXICITY.....: Cyfluthrin has been investigated in chronic feeding studies using two different strains of rats. In each study, cyfluthrin was administered for 2 years at dietary concentrations ranging from 50 to 450 ppm. Body weight gains were decreased at concentrations of 150 ppm and greater. Changes in clinical chemistries occurred at 450 ppm. In one of the studies, histopathology revealed a numerical increase in mammary gland adenocarcinomas at 450 ppm. This finding was not statistically significant when compared to the controls and is not considered to be compound-related. In each study, the overall NOEL was 50 ppm based on decreased body weight gains. In a 1 year feeding study, dogs were administered cyfluthrin at dietary concentrations of 50, 100, 360 or 650 ppm. Beginning on week 8, the high dose was reduced to 500 ppm for the remainder of the study due to severe clinical neurological symptoms. Body weights were decreased for animals of the high-dose. Neurological findings (gait abnormalities and postural reaction deficits) were observed at doses of 360 and greater. The NOEL was 100 ppm.

CARCINOGENICITY.....: Cyfluthrin was investigated for carcinogenicity in chronic studies using rats and mice at maximum levels of 450 and 800 ppm, respectively. There was no evidence of a carcinogenic potential observed in either species.

MUTAGENICITY.....: In vitro and in vivo mutagenicity studies have been conducted on BAY FCR 4545 technical, all of which are negative. Numerous in vitro and in vivo mutagenicity studies have been conducted on cyfluthrin, all of which are negative.

DEVELOPMENTAL TOXICITY: FCR 4545: In a developmental toxicity study, Bay FCR 4545 technical was administered orally to rats during gestation at doses of 3, 10 or 40 mg/kg. At the lethal and maternally toxic dose of 40 mg/kg, there was a decrease in fetal body weights and an increased incidence of skeletal findings. The NOELs for maternal and developmental toxicity were 3 and 10 mg/kg, respectively. Cyfluthrin: In developmental toxicity studies using rats, cyfluthrin was administered during gestation by oral gavage at doses ranging from 1 to 30 mg/kg. The overall NOEL from these studies for maternal toxicity was 3 mg/kg. No developmental effects were observed at any of the doses tested. In each study, the NOEL for developmental toxicity was equivalent to the highest dose tested. The NOELs for developmental toxicity for the initial study and the subsequent study were 30 and 10 mg/kg, respectively. Rabbits were administered cyfluthrin during gestation by oral gavage at doses ranging from 5 to 180 mg/kg. At maternally toxic levels, there was an increased incidence of post-implantation losses. The overall

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## 11. TOXICOLOGICAL INFORMATION (Continued)

NOEL derived from these studies for both maternal and developmental toxicity was 20 mg/kg. In an inhalation study, rats were exposed during gestation to cyfluthrin at aerosol concentrations of 0.46, 2.55 or 11.9 mg/m3 for 6 hours/day. NOELs for maternal and developmental toxicity were less than 0.46 and 0.46 mg/m3, respectively.

REPRODUCTION.....: In a reproduction study, cyfluthrin was administered to rats for 3 generations at dietary concentrations of 50, 150 and 450 ppm. Reproductive effects observed at parentally toxic levels included reductions in viability, lactation, litter size, feed consumption, and pup birth weights and body weight gains. Coarse tremors were observed in some offspring at 450 ppm. The NOEL for both parental and reproductive effects was 50 ppm. In another reproduction study, cyfluthrin was administered to rats for 2 generations at dictary concentrations of 50, 125 or 400 ppm. Coarse tremors occurring in conjunction with parental toxicity were observed in the offspring in the mid- and high-dose groups. Based on this finding, the neonatal NOEL was 50 ppm. The NOELs for parental and reproductive toxicity were 50 and 400 ppm, respectively.

NEUROTOXICITY .....: Numerous neurotoxicity studies have been conducted on cyfluthrin. Oral gavage studies using hens have indicated that at extremely high dose levels (5000 mg/kg), minimal nerve damage occurs. When rats were administered cyfluthrin daily at oral doscs of 40 to 80 mg/kg for 14 days, minimal nerve effects were seen. These effects were completely reversible within a 3 month recovery period. In dermal and inhalation studies which are relevant to field exposure, there was no evidence of delayed neurotoxicity in hens. In a special investigative study, litters of neonatal mice (10 days of age) and their mothers were exposed to aerosol concentrations of 5, 15, or 50 mg/m3 for 6.3 hours/day for 7 successive days. Motor activity was measured in the offspring at 4 months of age (approximately 3.5 months post-exposure). At 50 mg/m3, all of the offsprings died or were sacrificed in a moribund state following the first exposure. Mortalities were not observed at any of the other levels. Clinical symptoms were observed immediately after exposure in young mice at 15 mg/m3, and included decreased motility, temporary scratching, and tonic convulsions. There was an increase in motor activity in mice at 15 mg/m3. Histopathological investigations did not reveal any treatment-related findings in mice at the age of 4 months.

#### 12. ECOLOGICAL INFORMATION:

This product is extremely toxic to fish and aquatic invertebrates, and is highly toxic to bees. Bayer will provide a summary of specific data upon written request. As with any pesticide, this product should be used according to label directions and should be kept out of streams, lakes and other aquatic habitats of concern. In event of a spill emergency, call 1-800-414-0244.

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| 13. DISPOSAL CONSIDERATIONS   |
|---|
| WASTE DISPOSAL METHOD: Follow container label instructions for disposal of wastes generated during use in compliance with the FIFRA product label. In other situations, bury in an EPA approved landfill or burn in an incinerator approved for pesticide destruction.  EMPTY CONTAINER PRECAUTIONS: Do not reuse container without written permission and instructions from Bayer. Empty, clean and dispose in accordance with state and local laws. |
| 14. TRANSPORTATION INFORMATION:   |
| TECHNICAL SHIPPING NAME : beta-Cyfluthrin FREIGHT CLASS PACKAGE : Insecticides, NOI, NMFC 102100 PRODUCT LABEL TEMPO SC Ultra Premise Spray   |
| DOT (DOMESTIC SURFACE) HAZARD CLASS OR DIVISION: Non-Regulated  |
| It is not expected that a mist or vapor hazard would exist from the normal transportation of this liquid substance.   |
| TMO / IMDG CODE (OCEAN)  HAZARD CLASS DIVISION NUMBER: Non-Regulated  |
| ICAO / IATA (AIR)   |
| HAZARD CLASS DIVISION NUMBER: Non-Regulated   |
| 15. REGULATORY INFORMATION:   |
| OSHA STATUS: This product is hazardous under the criteria of<br>the Federal OSHA Hazard Communication Standard 29   |
| CFR 1910.1200.  ISCA STATUS: This product is exempt from TSCA Regulation under  FIFRA Section 3 (2)(B)(ii) when used as a   |
| pesticide.<br>CERCLA REPORTABLE QUANTITY: No components listed.   |

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#### 15. REGULATORY INFORMATION (Continued)

SARA TITLE III:

SECTION 302 EXTREMELY

HAZARDOUS SUBSTANCES ..: No components listed.

SECTION 311/312

HAZARD CATEGORIES....: Immediate Health Hazard

SECTION 313

TOXIC CHEMICALS.....: beta-Cyfluthrin (11.8%) - CAS # 68359-37-5

RCRA STATUS...... If discarded in its purchased form, this product would not be a hazardous waste either by listing or by characteristic. However, under RCRA, it is the responsibility of the product user to determine at the time of disposal, whether a material containing the product or derived from the product should be classified as a hazardous waste. (40 CFR 261.20-24)

#### 16. OTHER INFORMATION:

NFPA 704M RATINGS: Health Flammability Reactivity

0-Insignificant 1=Slight 2=Moderate 3=High 4=Extreme

Bayer's method of hazard communication is comprised of Product Labels and Material Safety Data Sheets. NFPA ratings are provided by Bayer as a customer service.

REASON FOR ISSUE..... Create new MSDS PREPARED BY..... V. C. Standart APPROVED BY..... D. C. Eberhart

TITLE..... Director Product Safety & Stewardship

APPROVAL DATE..... 05/06/2002

SUPERSEDES DATE..... Nonc MSDS NUMBER..... 29752

This information is furnished without warranty, expressed or implied, except that it is accurate to the best knowledge of Bayer Corporation. The data on this sheet relates only to the specific material designated herein. Bayer Corporation assumes no legal responsibility for use or reliance upon these

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It's Really Going Places.

#### Consumer Product Information

### TalstarOne™ Multi-Insecticide

Date Prepared: 23 April, 2004

#### 1. Product Information

Dilution of TalstarOne multi-insecticide, EPA Reg. # 279-3206 Chemical Family: Pyrethroid Insecticide

#### 2. Manufacturer Information

#### Manufacturer:

FMC Corporation Agricultural Products Group 1735 Market Street Philadelphia, PA 19103

#### Telephone Numbers:

Emergency Phone: (800)-331-3148 Spill Emergency: (800)-331-3148 Technical Information: (800)-321-1362

#### 3. Active Ingredient

CAS No.

Bifenthrin (0.12%) dilution

82657-04-3

Typical use rates are between 0.001% and 0.06%

#### 4. General Information and Precautions

Highly toxic to fish and aquatic organisms. Care should be used when spraying to avoid fish and reptile pets. Wash thoroughly with soap and water after handling.

#### 5. Health Hazard Information

General Health/Toxicity Information for 0.12% Suspension:
Acute Oral LD50 >20,000 mg/kg
Acute Dermal LD50 >10,000 mg/kg

This diluted material has a low oral and dermal toxicity, it is minimally irritating to the eyes and non-irritating to the skin. In rare occurrences, exposure to TalstarOne multi-insecticide end use dilution produces skin sensations such as burning, numbness or tingling during application. These sensations generally occur in the area of the body contacted by the diluted product and last for a few hours. This reaction, should it occur, produces some discomfort but does not result in skin damage.

#### 6. First Aid Procedures (Dilutions or Emulsions Only)

- Ingestion: Drink 1 or 2 glasses of water. Never give anything by mouth to an unconscious person. If any discomfort persists, obtain medical attention.
- Inhalation: Remove to fresh air. If breathing difficulty or discomfort occurs and persists, obtain medical attention.
- Dermal: Wash with plenty of soap and water
- Eye: Flush with plenty of water. Get medical attention if irritation occurs and persists
- Note to Physician or Emergency Personnel: TalstarOne multi-insecticide suspension contains a low concentration
  of the pyrethroid bifenthrin and primarily water. Should reversible skin sensations occur, skin salves have been
  found useful in reducing discomfort. Treatment is otherwise removal of exposure followed by symptomatic and
  supportive care.

#### 7. Fire and Hazards Information

The water-based suspension of TalstarOne multi-insecticide is not explosive or flammable.

#### 8. Environmental Hazards Information

This product is extremely taxic to fish. Do not contaminate water when disposing of equipment wash-waters.
 Do not apply directly to any body of water. Care should be used when spraying to avoid fish and reptiles.

#### 9. Storage and Disposal and Spill Information

- Keep out of reach of children. Store in a cool, dry place and avoid excess heat. Do not put concentrate or dilute material into food or drink containers.
- Isolate and post spill area. Keep animals and unprotected persons out of area. Keep emulsion out of streams
  and sewers. Dike to confine spills, and absorb with absorbent such as clay, sand or cat litter. Place in a DOT
  approved drum and contact appropriate regulatory agencies prior to disposal. To decontaminate spill area,
  tools and equipment, wash with a detergent/water mixture and properly dispose of solution as contaminated
  waste.

#### 10. Additional Information

 For safety and health information concerning the undiluted concentrate product, refer to the Material Safety Data Sheet for TalstarOne multi-insecticide.

Disclaimer: The information and recommendations contained herein are based upon data believed to be correct. However, no guarantee or warranty of any kind, expressed or implied, is made with respect to the information contained herein.

Status: Effective 4/2004

TalstarOne and FMC Logo - FMC trademarks

B0039

#### Bauer, Jaime

From:

Allen, John [John.Allen@iluka.com]

Sent:

Tuesday, November 06, 2007 4:40 PM

To:

Bauer, Jaime

Cc:

Jenkins, Ray

Subject: Corrected retention time page

Jaime.

Attached is the corrected retention time calculation page that you requested.

Sorry for the delay.

Regards,

John A. Allen

**Environmental Officer** 

Iluka Resources Inc

Office: (434)-348-4315 Cell: (804)-943-5611 john.allen@iluka.com

<<Retention\_time.pdf>>

**NOTICE** - Mail attachments have been compressed automatically and appear as .ZIP files. Should you be unable to access compressed attachments free compression software (e.g FreeZip) is readily available from the Internet.

This message and any attached files may contain information that is confidential and/or subject of legal privileged intended only for use by the intended recipient. If you are not the intended recipient or the person responsible for delivering the message to the intended recipient, be advised that you have received this message in error and that any dissemination, copying or use of this message or attachment is strictly forbidden, as is the disclosure of the information therein. If you have received this message in error please notify the sender immediately and delete the message. All care has been taken to ensure that this message and any attachments are virus free. No responsibility is accepted for any virus infections caused by receipt of this message.

# APPENDIX 4 RENTION TIME CALCULATIONS FOR RETENTION POND B

## Flow from the 1 hour / 25 year storm event was calculated using the Rational Method:

 $Q = C \times i \times A$ 

Q = Flow (cfs)

C = Runoff Coefficient

i = Inches of rain produced by the 1 hour / 25 year storm event

A = Acres of drainage area

As described in the Virginia Department of Conservation and Recreation's *Virginia Erosion and Sediment Control Handbook*, Third Edition, 1992.

#### The following values were used in the calculation:

The runoff coefficient (**C**) for the site is estimated to be **0.5**, based on the Land Use classification of Light Industrial.

Rainfall expected from the 1 hour / 25 year storm event (i) is approximately **2.75 inches.** This is based on the average of the City of Richmond and Greensville County curve values obtained from the DCR ESC Handbook.

The area (A) served by Retention Pond B is approximately 11.85 acres.

Therefore:

Q = 0.5 x 2.75 in x 11.85 ac = 16.3 cubic feet per second ⇒ 978 cubic feet per minute ⇒ 7,316 gallons per minute

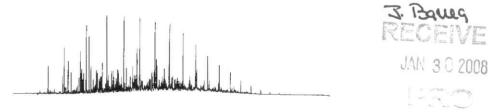
The volume of Retention Pond B is approximately **1.1 million gallons**.

Therefore, retention time for runoff produced by the 1 hour / 25 year storm is:

Retention Time = 1,100,000 gallons ÷ 7,316 gallons per minute = 150.4 minutes ⇒ **2.5 hours** 

## Primary Laboratories, Inc.

7423 Lee Davis Road • Mechanicsville, VA 23111 • Telephone (804) 559-9004 • Fax (804) 559-9306



## ANALYTICAL LABORATORY REPORT

24-Jan-08

ILUKA Resource, Inc. Attn: Kevin Rideout 12472 St. John Church Road Stony Creek, Virginia 23882

Date Received:

22-Jan-08

Date Sampled:

21-Jan-08

Work Order No:

0801151-01

| CHETICID.   | Outlan 002 |           |          |              |           |          |
|-------------|------------|-----------|----------|--------------|-----------|----------|
| Test        | Final      | Reporting | Units of | Standard     | Date      | Tech.    |
| Description | Result     | Limit     | Measure  | Methods (18) | Analyzed  | Initials |
| Metals      |            |           |          |              |           |          |
| Nickel      | <0.020     | 0.020     | mg/L     | 3030E/3120B  | 24-Jan-08 | HV       |
| 2           |            |           | 200      |              | at 13:38  |          |
| Zinc        | 0.049      | 0.010     | mg/L     | 3030E/3120B  | 24-Jan-08 | HV       |
|             |            |           |          |              | at 13:38  |          |

Note: Sample received in laboratory on ice.

Signature:

Parry L. Bragg

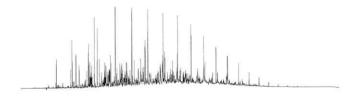
Laboratory Manager

\_Date://24/08

These analytical results are based upon materials provided by the client and are intended for the exclusive use of the client. These analytical results represent the best judgement of Primary Laboratories, Inc. Primary Laboratories, Inc. assumes no responsibility, express or implied, as to the interpretation of the analytical results contained in this report. This report is not to be reproduced except with the written approval of Primary Laboratories, Inc.

## Primary Laboratories, Inc.

7423 Lee Davis Road • Mechanicsville, VA 23111 • Telephone (804) 559-9004 • Fax (804) 559-9306



## ANALYTICAL LABORATORY REPORT

4-Jan-08

Iluka Resources, Inc Attn: Kevin Rideout

12472 St Johns Church Road Stoney Creek, VA 23882

Project:

Outfall 002 Renewal

Date Received:

31-Dec-07

Date Sampled:

31-Dec-07 0801001-01

Work Order No:

Outfall 002 (MSP)

| Client ID:  | Outfall 002 (MS |           |          |                                    |                      |          |
|-------------|-----------------|-----------|----------|------------------------------------|----------------------|----------|
| Test        | Final           | Reporting | Units of | Standard                           | Date                 | Tech.    |
| Description | Result          | Limit     | Measure  | Methods (18)                       | Analyzed             | Initials |
| Ammonia     | 0.09            | 0.01      | mg/L     | 4500NH <sub>3</sub> F              | 3-Jan-08             | NA       |
| Alkalinity  | 1.9             | 0.1       | mg/L     | 2320 B                             | at 8:00<br>3-Jan-08  | NA       |
| TKN         | 6.8             | 0.5       | mg/L     | 4500 N/ <sub>NH3</sub>             | at 12:00<br>3-Jan-08 | NA       |
| Nitrite     | 0.055           | 0.005     | mg/L     | 4500 NO <sub>2</sub> -B            | at 9:30<br>2-Jan-08  | NA       |
| Nitrate     | 1.44            | 0.05      | mg/L     | 4500 NO <sub>3</sub> -E            | at 9:15<br>2-Jan-08  | NA       |
| Sulfate     | 232.10          | 2.0       | mg/L     | 4500SO <sub>4</sub> <sup>2</sup> E | at 15:45<br>4-Jan-08 | NA       |
|             |                 |           |          |                                    | at 13:30             |          |

# Primary Laboratories, Inc. Results

4-Jan-08

Date Sampled: Work Order No:

31-Dec-07

Client ID:

0801001-01 Outfall 002 (MSP)

| Cilent ID.         | Outrail 002 (MS | P)         |          |              |          |          |
|--------------------|-----------------|------------|----------|--------------|----------|----------|
| Test               | Final           | Reporting  | Units of | Standard     | Date     | Tech.    |
| Description        | Result          | Limit      | Measure  | Methods (18) | Analyzed | Initials |
| Metals             |                 |            |          |              |          |          |
| Arsenic            | <50             | 50         | ug/L     | 3030E/3120B  | 4-Jan-08 | HV       |
| Name of the second |                 |            |          |              | at 11:07 |          |
| Barium             | 41              | 20         | ug/L     | 3030E/3120B  | 4-Jan-08 | HV       |
|                    |                 |            |          |              | at 11:07 |          |
| Cadmium            | <10             | 10         | ug/L     | 3030E/3120B  | 4-Jan-08 | HV       |
|                    |                 |            |          |              | at 11:07 |          |
| Chromium           | 23              | 20         | ug/L     | 3030E/3120B  | 4-Jan-08 | HV       |
| 1 Dec. 20          | 200000 88       |            |          |              | at 11:07 |          |
| Iron               | 604             | 50         | ug/L     | 3030E/3120B  | 4-Jan-08 | HV       |
| îî                 |                 |            |          |              | at 11:07 |          |
| Lead               | <50             | 50         | ug/L     | 3030E/3120B  | 4-Jan-08 | HV       |
| **                 | 27.2            |            |          |              | at 11:07 |          |
| Mercury            | <0.2            | 0.2        | ug/L     | 3112 B       | 3-Jan-08 | HV       |
| Bro Mariano        |                 | 111 212 22 | DEV      |              | at 13:30 |          |
| Potassium          | 7,640           | 1,000      | ug/L     | 3030E/3120B  | 4-Jan-08 | HV       |
| 0.1.               |                 |            |          |              | at 11:07 |          |
| Selenium           | <50             | 50         | ug/L     | 3030E/3120B  | 4-Jan-08 | HV       |
| 0:1                |                 |            | 123      | 20.          | at 11:07 |          |
| Silver             | <20             | 20         | ug/L     | 3030E/3120B  | 4-Jan-08 | HV       |
| Cadium             | 4.050           |            |          |              | at 11:07 |          |
| Sodium             | 4,050           | 1,000      | ug/L     | 3030E/3120B  | 4-Jan-08 | HV       |
| Titonium           | .00             |            | 707      |              | at 11:07 |          |
| Titanium           | <20             | 20         | ug/L     | 3030E/3120B  | 4-Jan-08 | HV       |
|                    |                 |            |          |              | at 11:07 |          |

NOTE: Samples received in laboratory on ice.

Signature:

Parry L. Bragg Laboratory Manager Date:

1-4-08

These analytical results are based upon materials provided by the client and are intended for the exclusive use of the client. These analytical results represent the best judgement of Primary Laboratories, Inc. Primary Laboratories, Inc. assumes no responsibility, express or implied, as to the interpretation of the analytical results contained in this report. This report is not to be reproduced except with the written approval of Primary Laboratories, Inc.



## COMMONWEALTH of VIRGINIA

## DEPARTMENT OF ENVIRONMENTAL QUALITY

L. Preston Bryant, Jr. Secretary of Natural Resources PIEDMONT REGIONAL OFFICE 4949-A Cox Road, Glen Allen, Virginia 23060 (804) 527-5020 Fax (804) 527-5106 www.deq.virginia.gov

David K. Paylor Director

Gerard Seeley, Jr. Regional Director

TO: Curt Linderman
FROM: Jaime Bauer
DATE: November 9, 2007

SUBJECT: Waiver Request for VA0090981 – Iluka Resources Inc, Mineral Separation Plant

COPIES: File (R/G, right)

Iluka Resources Inc. is requesting a waiver from composite sampling for all required parameters for the effluent testing from Retention Pond 3 (Outfall 002) and asks that grab samples be accepted to satisfy the VPDES permit application requirements. They are also requesting that previous sampling data greater than 3 years old be accepted to satisfy the requirements of Form 2F Section VII Part C.

#### Reasons:

The permit application for Storm Water Discharges Associated with Industrial Activity (Form 2F) automatically allows the use of grab samples in lieu of composite samples from holding ponds with retention periods greater than 24 hours. The retention period at Pond 3 is estimated to be 2.5 hours; therefore they do not qualify for the automatic waiver. Documentation provided by the facility shows a submersible pump located in the southwest corner of the pond that recirculates the water within the pond prior to discharge. The facility believes that the mixing creates an effluent of consistent quality therefore a grab sample would provide a representative sample of the effluent.

Iluka is also requesting that test data from 2000 be accepted to satisfy the permit application requirements in Form 2F Section VII Part C so that the permit reissuance can proceed. The facility has stated that the 2000 data is still representative of facility operations. Form 2F provides that past data can be used if it is less than 3 years old, the samples are representative of the present discharge, and all requirements are met. Because the data is from 2000, it does not meet the requirements listed in the permit application. The waiver is being requested due to lack of qualifying storm events over the past several months and the current permit will expire on January 26, 2008.

I recommend that the grab samples be accepted as representative samples for the purpose of satisfying the permit application requirements in Form 2F.

The facility has submitted a plan to join Retention Pond 3 and the Sediment Pond by breaching a berm currently separating them. The outfall from the Sediment Pond will be closed. Following that construction, staff intends to require sampling at Outfall 002 which should confirm the data reported in the permit renewal application.

I recommend that the 2000 sampling results be accepted as adequate for the permit renewal application.

|           | Y         | Approved                               |                    |                     | Denied          |
|-----------|-----------|--|--------------------|---------------------|-----------------|
| Comments: | reguiring | mmended,<br>Outfall 00.<br>ner clause. | for this paint ago | le only<br>rech) 's | hould include a |
| 4         | Signature |  |                    | Da                  | 11/13/87<br>te  |



October 29, 2007

Ms. Jaime Bauer DEQ-PRO 4949 A Cox Road Glen Allen, VA 23060

RE: Request for Composite Sampling Wavier and Statement Regarding Validity of Data.

Ms. Bauer:

lluka would like to request a waiver for composite sampling requirements in Form 2F for outfall 002. While detention time in Retention Pond 3 is less than 24 hours for the 25 year storm event, it is my belief that the grab samples taken from this outfall are representative of the pond's overall water quality. This is due to use of a submersible pump to recirculate and mix the water within the pond. The pump is located in the southwest corner of the pond and discharges toward the north at a rate of 200 to 300 gpm. This provides adequate mixing of the pond's water to ensure a discharge of relatively consistent quality. Please see the enclosed figure depicting the placement of the submersible pump, its discharge point and the probable circulation patterns within the pond.

Iluka would also like to request that the data from 2000 which was submitted in Part VII Section C of Form 2F be accepted for the purposes of the renewal. I believe that there have been no materials handling or process changes at the site which would result in significant variations from the 2000 data. Iluka proposes to resample for these parameters at the first opportunity.

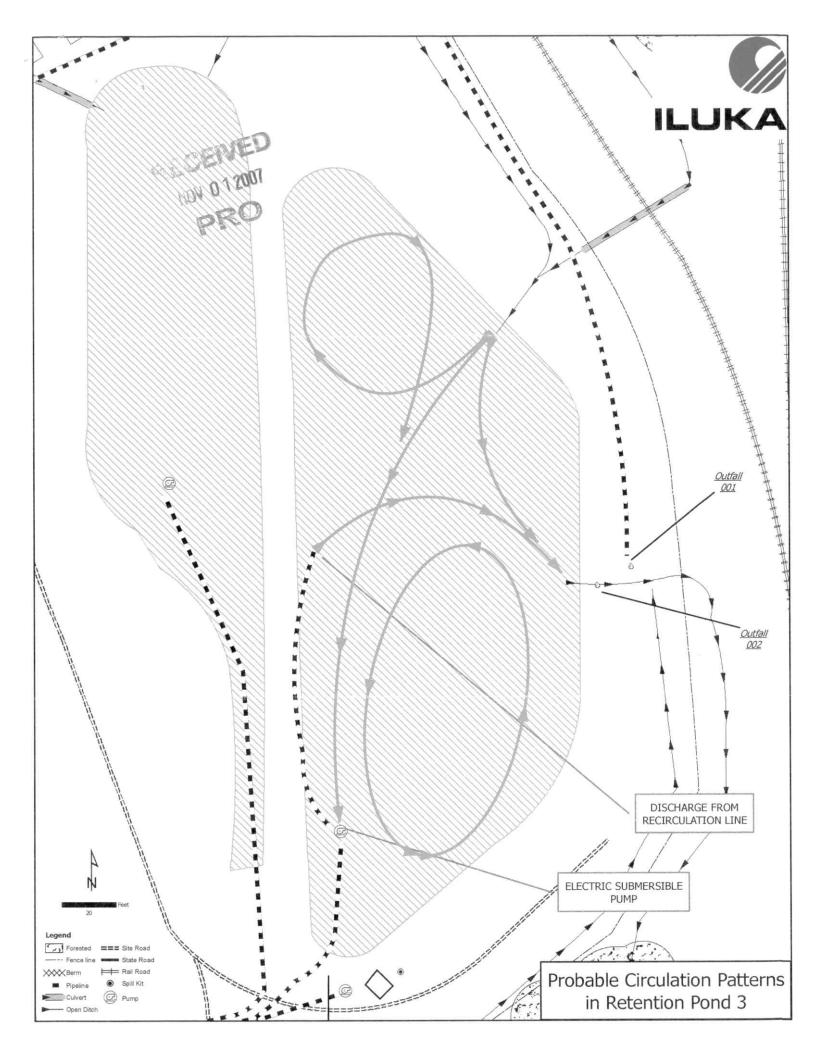
Your consideration of these requests is greatly appreciated.

Sincerely,

John A. Allen

**Environmental Officer** 

Enclosure





USPS - Certified, Return Receipt Requested

April 17, 2008

Ms. Jaime Bauer VPDES/VPA Permit Writer Department of Environmental Quality Piedmont Regional Office 4949-A Cox Road Glen Allen, VA 23060 RECEIVED

APR 2 1 2008

PRO

RE: Application for Process Water Discharge Outfall 004

Dear Ms. Bauer:

During the renewal period for VPDES Permit Number 0090981, Iluka has submitted two proposed amendments for consideration by DEQ. These proposed amendments are:

- Removal of the berm separating Retention Pond 3 from the Sediment Pond and sealing of the Sediment Pond outfall
- Addition of the Admin Stormwater Pond and it's outfall to the site's Individual Permit as outfall 003

Both of the above referenced ponds and their outfalls are currently covered under the site's General Stormwater Permit, VPDES #VAR051217. These proposed changes will remove the need for the site to maintain this permit.

While VA0090981 is open for renewal and consideration of these amendments, Iluka would like to propose the addition of outfall 004 to serve as a discharge point for excess water from the site's Process Water Ponds. A full description of the proposed change is outlined in the Narrative document attached with Form 2C.

If you have any questions or need additional information or clarification regarding this proposal please contact me at the numbers listed below or via email at john.allen@iluka.com.

Regards,

John A. Allen

Environmental Officer

804-943-5611 (C) 434-348-4315 (O) EPA I.D. NUMBER (copy from Item 1 of Form 1)

Please print or type in the unshaded areas only.

VA0090981

Form Approved. OMB No. 2040-0086. Approval expires 3-31-98.

FORM 2C **NPDES**  U.S. ENVIRONMENTAL PROTECTION AGENCY APPLICATION FOR PERMIT TO DISCHARGE WASTEWATER

EXISTING MANUFACTURING, COMMERCIAL, MINING AND SILVICULTURE OPERATIONS

Consolidated Permits Program

| A. OUTFALL NUMBER | E       | B. LATITUDE |         | C. LONGITUDE |         |         |                           |
|-------------------|---------|-------------|---------|--------------|---------|---------|---------------------------|
| (list)            | 1. DEG. | 2. MIN.     | 3. SEC. | 1. DEG.      | 2. MIN. | 3. SEC. | D. RECEIVING WATER (name) |
| 004 (proposed)    | 36      | 58          | 10.41   | 77           | 24      | 11.75   | GALLEY SWAMP              |
|                   |         |             |         |              |         |         | see Fig. 1 for location   |
|                   |         |             |         |              |         |         |                           |
|                   |         |             |         |              |         |         |                           |
|                   |         |             |         |              |         |         |                           |

#### II. FLOWS, SOURCES OF POLLUTION, AND TREATMENT TECHNOLOGIES

- A. Attach a line drawing showing the water flow through the facility. Indicate sources of intake water, operations contributing wastewater to the effluent, and treatment units labeled to correspond to the more detailed descriptions in Item B. Construct a water balance on the line drawing by showing average flows between intakes, operations, treatment units, and outfalls. If a water balance cannot be determined (e.g., for certain mining activities), provide a pictorial description of the nature and amount of any sources of water and any collection or treatment measures. Please See APPENDIX 1, FIGURE 3.
- B. For each outfall, provide a description of: (1) All operations contributing wastewater to the effluent, including process wastewater, sanitary wastewater, cooling water, and storm water runoff; (2) The average flow contributed by each operation; and (3) The treatment received by the wastewater. Continue on additional sheets if

| 1. OUT-            | 2. OPERATION(S) CONTRIBUTING FLOW       |                                    | 3. TREATMENT   |                                  |  |  |
|--------------------|---|------------------------------------|----------------|----------------------------------|--|--|
| FALL<br>NO. (list) | u. o. u                                 | b. AVERAGE FLOW<br>(include units) | a. DESCRIPTION | b. LIST CODES FROM<br>TABLE 2C-1 |  |  |
| 004                | Mineral sands processing and handling.  | NA*                                | See Document 1 | 1-0, 1-U 4-A, 4-C                |  |  |
| (propo<br>sed)     |   |                                    |                |                                  |  |  |
|                    |   |                                    |                |                                  |  |  |
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|                    |   |                                    |                |                                  |  |  |
|                    |   |                                    |                |                                  |  |  |
| OFFICIAL           | USE ONLY (effluent guidelines sub-cates | rories)                            |                |                                  |  |  |

CONTINUED FROM THE FRONT C. Except for storm runoff, leaks, or spills, are any of the discharges described in Items II-A or B intermittent or seasonal? YES (complete the following table) NO (go to Section III) 3. FREQUENCY 4. FLOW B. TOTAL VOLUME a. DAYS PER WEEK 2. OPERATION(s) CONTRIBUTING FLOW a. FLOW RATE (in mgd) b. MONTHS (specify with 1. OUTFALL (specify average) PER YEAR C. DURATION 1. LONG TERM 1. LONG TERM 2. MAXIMUM 2. MAXIMUM DAILY NUMBER (/isr) (specify average (list) DAILY AVERAGE (in days) Mineral sands processing and 004 1 mgd 1.4 mgd (est.) 1 mgd (est.) 1.4 mgd (est.) 1 (est.) (proposed) average (est.) average (est.) (est.) III. PRODUCTION A. Does an effluent guideline limitation promulgated by EPA under Section 304 of the Clean Water Act apply to your facility? YES (complete Item III-B) NO (go to Section IV) B. Are the limitations in the applicable effluent guideline expressed in terms of production (or other measure of operation)? YES (complete Item III-C) ✓ NO (go to Section IV) C. If you answered "yes" to Item III-B, list the quantity which represents an actual measurement of your level of production, expressed in the terms and units used in the applicable effluent guideline, and indicate the affected outfalls. 1. AVERAGE DAILY PRODUCTION 2. AFFECTED OUTFALLS c. OPERATION, PRODUCT, MATERIAL, ETC. (list outfall numbers) a. QUANTITY PER DAY b. UNITS OF MEASURE (specify) na na na IV. IMPROVEMENTS A. Are you now required by any Federal, State or local authority to meet any implementation schedule for the construction, upgrading or operations of wastewater treatment equipment or practices or any other environmental programs which may affect the discharges described in this application? This includes, but is not limited to, permit conditions, administrative or enforcement orders, enforcement compliance schedule letters, stipulations, court orders, and grant or loan conditions. NO (go to Item IV-B) YES (complete the following table) 1. IDENTIFICATION OF CONDITION. 2. AFFECTED OUTFALLS 4. FINAL COMPLIANCE DATE 3. BRIEF DESCRIPTION OF PROJECT AGREEMENT, ETC. a. NO b SOURCE OF DISCHARGE a REQUIRED b. PROJECTED na na B. OPTIONAL: You may attach additional sheets describing any additional water pollution control programs (or other environmental projects which may affect your discharges) you now have underway or which you plan. Indicate whether each program is now underway or planned, and indicate your actual or planned schedules for construction MARK "X" IF DESCRIPTION OF ADDITIONAL CONTROL PROGRAMS IS ATTACHED

EPA I.D. NUMBER (copy from Item 1 of Form 1) VA0090981

#### CONTINUED FROM PAGE 2

| V. INTAKE AND EFFLUENT CHARACTER  | RISTICS CONTROL OF THE PARTY OF |  |  |  |  |  |
|---|--|--|--|--|--|--|
| A, B, & C: See instructions before proceeding – Complete one set of tables for each outfall – Annotate the outfall number in the space provided. NOTE: Tables V-A, V-B, and V-C are included on separate sheets numbered V-1 through V-9.   |  |  |  |  |  |  |
| D. Use the space below to list any of the pollutants listed in Table 2c-3 of the instructions, which you know or have reason to believe is discharged or may be discharged from any outfall. For every pollutant you list, briefly describe the reasons you believe it to be present and report any analytical data in your possession. |  |  |  |  |  |  |
| 1. POLLUTANT  | 2. SOURCE  | 1. POLLUTANT   | 2. SOURCE                              |  |  |  |
| None  | na   |  |  |  |  |  |
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| VI. POTENTIAL DISCHARGES NOT COV  |  |  |  |  |  |  |
| Is any pollutant listed in Item V-C a substa  YES (list all such pollutants   | nce or a component of a substance which you  | ou currently use or manufacture as an intern<br>NO (go to Item VI-B) | nediate or final product or byproduct? |  |  |  |
| na  |  |  |  |  |  |  |
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EPA Form 3510-2C (8-90)

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|         |  |  |

| VII. BIOLOGICAL TOXICITY TESTING DATA  | LANGE CONTRACTOR OF THE SAME AND  |   | 起音的 医腺素 计正数图数                                 |
|--|---|---|---|
| Do you have any knowledge or reason to beli<br>relation to your discharge within the last 3 year   | eve that any biological test for acute or chronic toxici  | ty has been made on any of your dis   | scharges or on a receiving water in           |
| YES (identify the test(s) and des  |   | NO (go to Section VIII)   |   |
| No Whole Effluent Toxicity (W<br>taken from outfall 001 as a c<br>Pollution Discharge Eleminati  | MET) testing has been proformed on condition of Virginia Department of on Program (VPDES) permit VA009098 or, VA for testing. The results of  | proposed outfall 004. W<br>Environmental Quality'   | s (DEQ) Virginia<br>submitted to Coastal      |
| VIII. CONTRACT ANALYSIS INFORMATION  |   |   | positive day of the stage of the stage of the |
| Were any of the analyses reported in Item V  | performed by a contract laboratory or consulting firm   | ?   |   |
| YES (list the name, address, and each such laboratory or fire  | d telephone number of, and pollutants analyzed by,<br>m below)  | NO (go to Section IX)   |   |
| A. NAME  | B. ADDRESS  | C. TELEPHONE (area code & no.)  | D. POLLUTANTS ANALYZED (list)                 |
| Primary Laboratories   | 7423 Lee Davis Road<br>Mechanicsville, VA 23111   | 804-559-9004  | See Attached Lab Report Sheets.               |
|  |   |   |   |
| IX CERTIFICATION   |   |   |   |
| I certify under penalty of law that this docum<br>qualified personnel properly gather and eve<br>directly responsible for gathering the informa<br>are significant penalties for submitting false.<br>A. NAME & OFFICIAL TITLE (type or print) | nent and all attachments were prepared under my dir<br>aluate the information submitted. Based on my inqu<br>ation, the information submitted is, to the best of my<br>information, including the possibility of fine and impri | iry of the person or persons who<br>knowledge and belief, true, accurate<br>sonment for knowing violations.<br>B. PHONE NO. (area code & no.) | manage the system or those persons            |
| qualified personnel properly gather and evalurectly responsible for gathering the information  | aluate the information submitted. Based on my inquation, the information submitted is, to the best of my information, including the possibility of fine and impris  | uiry of the person or persons who<br>knowledge and belief, true, accurate<br>sonment for knowing violations.                                  | manage the system or those persons            |

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| VII. BIOLOGICAL TOXICITY TESTING DATA   | <b>在1987年,1987年,1987年,1987年</b>  |   | <b>经验证的</b> 中的                          |
|---|--|---|---|
| Do you have any knowledge or reason to beli<br>relation to your discharge within the last 3 year  | eve that any biological test for acute or chronic toxici<br>ars?   | y has been made on any of your dis  | scharges or on a receiving water in     |
| YES (identify the test(s) and des   | cribe their purposes below)  | NO (go to Section VIII)   |   |
| taken from outfall 001 as a c<br>Pollution Discharge Eleminati  | ET) testing has been proformed on ondition of Virginia Department of on Program (VPDES) permit VA009098 r, VA for testing. The results of  | Environmental Quality'<br>1. These samples were s   | s (DEQ) Virginia<br>ubmitted to Coastal |
|   |  |   |   |
| VIII. CONTRACT ANALYSIS INFORMATION   |  | and the production of the second | Adams in the party of the party of      |
| Were any of the analyses reported in Item V   | performed by a contract laboratory or consulting firm  | )   |   |
| YES (list the name, address, and each such laboratory or fire   | d telephone number of, and pollutants analyzed by,<br>m below)   | NO (go to Section LX)   |   |
| A. NAME   | B. ADDRESS   | C. TELEPHONE (area code & no.)  | D. POLLUTANTS ANALYZED (list)           |
| Primary Laboratories  | 7423 Lee Davis Road<br>Mechanicsville, VA 23111  | 804-559-9004  | See Attached Lab Report<br>Sheets.      |
|   |  |   |   |
| IX. CERTIFICATION   |  |   |   |
| I certify under penalty of law that this docum<br>qualified personnel properly gather and evi<br>directly responsible for gathering the informa<br>are significant penalties for submitting false | ent and all attachments were prepared under my dir<br>aluate the information submitted. Based on my inqu<br>ation, the information submitted is, to the best of my<br>information, including the possibility of fine and impri | iry of the person or persons who i<br>knowledge and belief, true, accurate                                    | manage the system or those persons      |
| A. NAME & OFFICIAL TITLE (type or print)  Allan R. Sale, President, Ilu:  | ka US Operations   | B. PHONE NO. (area code & no.)<br>(434) 348-4300  |   |
|   |  | D. DATE SIGNED  |   |
| C. SIGNATURE Alla A Ja  | ·le  | 4/24/08   | PECEIVED.                               |
| EPA Form 3510-2C (8-90)   | PAGE 4 of 4  |   | 1 Charles Williams                      |

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PLEASE PRINT OR TYPE IN THE UNSHADED AREAS ONLY. You may report some or all of this information on separate sheets (use the same format) instead of completing these pages. SEE INSTRUCTIONS.

V. INTAKE AND EFFLUENT CHARACTERISTICS (continued from page 3 of Form 2-C)

YA00909.1 You may report some or all of this information PA LD. NUMBER (copy from Item I of Form I) yathese pages.

PART A -You must provide the results of at least one analysis for every pollutant in this table. Complete one table for each outfall. See instructions for additional details.

OUTFALL NO.

b. NO. OF ANALYSES (2) MASS 4. INTAKE a. LONG TERM AVERAGE VALUE (1) CONCENTRATION VALUE VALUE VALUE b. MASS STANDARD UNITS 3. UNITS (specify if blank) ပ ပ္ a. CONCENTRATION mg/L mg/L mg/L mg/L mg/L d. NO. OF ANALYSES 138 Н  $\vdash$ -Н  $\vdash$ 0 52 36 (2) MASS c. LONG TERM AVRG. VALUE (if available) 15.5 31.8 na (1) CONCENTRATION VALUE VALUE VALUE 2. EFFLUENT b. MAXIMUM 30 DAY VALUE (if available) MAXIMUM 6.17 (2) MASS 17.4 33.7 na (1) CONCENTRATION MINIMUM 5.22 VALUE VALUE VALUE MAXIMUM 9.07 a. MAXIMUM DAILY VALUE (2) MASS 20.8 36.2 (1) CONCENTRATION na 30.5 <3.0 11.5 MINIMUM 4.24 1.33 3.31 VALUE VALUE VALUE c. Total Organic Carbon a. Biochemical Oxygen b. Chemical OxygenDemand (COL) 1. POLLUTANT d. Total Suspended Solids (TNS) e. Ammonia (as N) g. Temperature h. Temperature Demand (BOD) summer) (winter) f. Flow i. pH

Mark "X" in column 2-a for each pollutant you know or have reason to believe is present. Mark "X" in column 2-b for each pollutant you believe to be absent. If you mark column 2a for any pollutant which is limited either directly, or indirectly but expressly, in an effluent limitations guideline, you must provide the results of at least one analysis for that pollutant. For other pollutants for which you mark column 2a, you must provide quantitative data or an explanation of their presence in your discharge. Complete one table for each outfall. See the instructions for additional details and requirements. PART B-

|                                | 2. MARK "X"         | *X "X"   |                        |          | 3.                               | 3. EFFLUENT |  |            |          | 4. UNITS         | TS      | 5. INT                                      | 5. INTAKE (optional) | //                |
|--------------------------------|---------------------|----------|------------------------|----------|----------------------------------|-------------|--|------------|----------|------------------|---------|---|----------------------|-------------------|
| 1. POLLUTANT<br>AND            | m                   | ģ        | a. MAXIMUM DAILY VALUE | LY VALUE | b. MAXIMUM 30 DAY (if available) | DAY VALUE   | b. MAXIMUM 30 DAY VALUE c. LONG TERM AVRG. VALUE ((favailable) | VRG. VALUE | 9        |                  |         | a. LONG TERM AVERAGE<br>VALUE               | AVERAGE              | L<br>C            |
| (if available)                 | BELIEVED<br>PRESENT | BELIEVED | (1)<br>CONCENTRATION   | (2) MASS | (1)<br>CONCENTRATION             | (2) MASS    | (1)<br>CONCENTRATION   | (2) MASS   | ANALYSES | a. CONCENTRATION | b. MASS | a. CONCEN-<br>TRATION b. MASS CONCENTRATION | (2) MASS             | (2) MASS ANALYSES |
| a. Bromide<br>(24959-67-9)     |                     | ×        |                        |          |                                  |             |  |            |          |                  |         |   |                      |                   |
| b. Chlorine, Total<br>Residual | ×                   |          | 24.1                   |          |                                  |             |  |            | Н        | mg/L             |         |   |                      |                   |
| c. Color                       |                     | ×        |                        |          |                                  |             |  |            |          |                  |         |   |                      |                   |
| d. Fecal Coliform              | X                   |          | < 2                    |          |                                  |             |  |            | п        | MPN/100mL        |         |   |                      |                   |
| e. Fluoride<br>(16984-48-8)    |                     | ×        |                        |          |                                  |             | la la  |            |          |                  |         |   |                      |                   |
| f. Nitrate-Nitrite (as N)      |                     | ×        |                        |          |                                  |             |  |            |          |                  |         |   |                      |                   |

EPA Form 3510-2C (8-90)

PAGE V-1

CONTINUE ON REVERSE

from Standard Methods (18) М 4500CL Note: Chlorides data obtained using procedure

supplied for Total Residual Chlorine values.

ITEM V-B CONTINUED FROM FRONT

| TEM V=B CONTINUED FROM FROM                         | INOLD IN    | 1000     |                        |           |  |             |   |                       |                  |         |                               |                      |                       |
|---|-------------|----------|------------------------|-----------|--|-------------|---|-----------------------|------------------|---------|-------------------------------|----------------------|-----------------------|
| 1   | 2. MARK "X" | -X- X-   |                        |           | 3.                                     | 3. EFFLUENT |   |                       | 4. UNITS         | LS      | 5. INTA                       | 5. INTAKE (optional) |                       |
| 1. POLLUTANT<br>AND                                 | ë           | á        | a. MAXIMUM DAILY VALUE | ILY VALUE | b. MAXIMUM 30 DAY VALUE (if available) | DAY VALUE   | c. LONG TERM AVRG. VALUE (if available) |                       |                  |         | a. LONG TERM<br>AVERAGE VALUE |                      |                       |
| _   | BELIEVED    | BELIEVED | (1)<br>CONCENTRATION   | (2) MASS  | (1)<br>CONCENTRATION                   | (2) MASS    | (1)<br>CONCENTRATION                    | d. NO. OF<br>ANALYSES | a. CONCENTRATION | b. MASS | (1)<br>CONCENTRATION          | ASS                  | b. NO. OF<br>ANALYSES |
| g. Nitrogen,<br>Total Organic (478                  |             | ×        |                        |           |  |             |   |                       |                  |         |                               |                      |                       |
| h. Oil and<br>Grease                                |             | ×        |                        |           |  |             |   |                       |                  |         |                               |                      |                       |
| i. Phosphorus<br>(as P), Total<br>(7723-14-0)       |             | ×        |                        |           |  |             |   |                       |                  |         |                               |                      |                       |
| j. Radioactivity                                    |             |          |                        |           |  |             |   |                       |                  |         |                               |                      |                       |
| (1) Alpha, Total                                    | ×           |          | 17.8 ± 2.3             |           |  |             |   | Н                     | pci/L            |         |                               |                      |                       |
| (2) Beta, Total                                     | ×           |          | 13.2 ± 1.0             |           |  |             |   | н                     | pci/L            |         |                               |                      |                       |
| (3) Radium,<br>Total                                |             | ×        |                        |           |  |             |   |                       |                  |         |                               |                      |                       |
| (4) Radium 226,<br>Total                            |             | ×        |                        |           |  |             |   |                       |                  |         |                               |                      |                       |
| k. Sulfate<br>(as SO <sub>J</sub> )<br>(14808-79-8) |             | ×        |                        |           |  |             |   |                       |                  | 1,      |                               |                      |                       |
| I. Sulfide (as S)                                   |             | ×        |                        |           |  |             |   |                       |                  |         |                               |                      |                       |
| m. Sulfite (as SO <sub>3</sub> ) (14265-45-3)       |             | ×        |                        |           |  |             |   |                       |                  |         |                               |                      |                       |
| n. Surfactants                                      |             | ×        |                        |           |  |             |   |                       |                  |         |                               |                      |                       |
| o. Aluminum,<br>Total<br>(7429-90-5)                |             | ×        |                        |           |  |             |   |                       |                  |         |                               |                      |                       |
| p. Barium, Total<br>(7440-39-3)                     |             | ×        |                        |           |  |             |   |                       |                  |         |                               |                      |                       |
| q. Boron, Total<br>(7440-42-8)                      |             | ×        |                        |           |  |             |   |                       |                  |         |                               |                      |                       |
| r. Cobalt, Total<br>(7440-48-4)                     |             | ×        |                        |           |  |             |   |                       |                  |         |                               |                      |                       |
| s. Iron, Total<br>(7439-89-6)                       | ×           |          | <0.050                 |           |  |             |   | 1                     | mg/L             |         |                               |                      |                       |
| t. Magnesium,<br>Total<br>(7439-95-4)               |             | ×        |                        |           |  |             |   |                       |                  |         |                               |                      |                       |
| u. Molybdenum,<br>Total<br>(7439-98-7)              |             | ×        |                        |           |  |             |   |                       |                  |         |                               |                      |                       |
| v. Manganese;<br>Total<br>(7439-96-5)               |             | ×        |                        |           |  |             |   |                       |                  |         | [8]                           |                      |                       |
| w. Tin. Total<br>(7440-31-5)                        |             | ×        |                        |           |  |             |   |                       |                  |         |                               |                      |                       |
| x. Titanium,<br>Total<br>(7440-32-6)                | X           |          | <0.020                 |           |  |             |   | н                     | mg/L             |         |                               |                      |                       |
| EPA Form 3510-2C (8-90)                             | 2C (8-90)   |          |                        |           |  |             | PAGE V-2                                |                       |                  |         | 00                            | CONTINUE ON PAGE V-3 | V PAGE V-3            |

| EPA I.D. NUMBER (copy from Item 1 of Form 1) | OUTFALL NUMBER |
|--|----------------|
| VA0090981                                    | 004            |

provide the results of at least one analysis for that pollutant. If you mark column 2b for any pollutant, you must provide the results of at least one analysis for that pollutant. If you mark column 2b for acrolein, acrylonitrile, 2,4 dinitrophenol, or 2-methyl-4, 6 dinitrophenol, you must provide the results of at least one analysis for each of these pollutants which you know or have reason to believe that you discharge in concentrations of 100 ppb or greater. Otherwise, for pollutants for which you mark column 2b, you must either submit at least one analysis or briefly describe the reasons the pollutant is expected to be discharged. Note that there are 7 pages to this part; please review each carefully. Complete one table (all 7 pages) for each outfall. See instructions for PART C - If you are a primary industry and this outfall contains process wastewater, refer to Table 2c-2 in the instructions to determine which of the GCMS fractions you must test for. Mark "X" in column 2-a for all such GCMS fractions that apply to your industry and for ALL toxic metals, cyanides, and total phenols. If you are not required to mark column 2-a (secondary industries, nonprocess wastewater outfalls, and nonrequired GCMS fractions), mark "X" in column 2-b for each pollutant you know or have reason to believe is present. Mark "X" in column 2-c for each pollutant you believe is absent. If you mark column 2a for any pollutant, you must CONTINUED FROM PAGE 3 OF FORM 2-C

ANALYSES 5. INTAKE (optional) (2) MASS a. LONG TERM AVERAGE VALUE (1) CONCENTRATION b. MASS 4. UNITS a. CONCENTRATION mg/L mg/L /Bm /Bu /Bu /Bm mg/ d. NO. OF ANALYSES Н -+H $\dashv$ -(2) MASS c. LONG TERM AVRG. VALUE (if available) (1) CONCENTRATION b. MAXIMUM 30 DAY VALUE (If available) (2) MASS 3. EFFLUENT CONCENTRATION a. MAXIMUM DAILY VALUE (2) MASS DESCRIBE RESULTS CONCENTRATION 050 <0.020 <0.020 <0.050 <0.002 <0.010 0.020 0 > G. BELIEVED ABSENT × × × × × × additional details and requirements. AND TOTAL PHENOLS BELIEVED E 2. MARK "X TESTING REQUIRED × × × × × × × METALS, CYANIDE, Total Antimony, Total 3M. Beryllium, Total (7440-41-7) 12M. Thallium, Total (7440-28-0) CAS NUMBER 11M. Silver, Total Dioxin (1764-01-6) 1. POLLUTANT Total 6M. Copper, Total (7440-50-8) 10M. Selenium, Total (7782-49-2) Total (7440-47-3) 13M. Zinc, Total 9M. Nickel, Total chlorodibenzo-P-4M. Cadmium, T (7440-43-9) Fotal (57-12-5) 7M. Lead, Total (7439-92-1) 5M. Chromium. 8M. Mercury, T (7439-97-6) 14M. Cyanide 15M. Phenois 2M. Arsenic, T (7440-38-2) 2,3,7,8-Tetra-7440-66-6) 7440-22-4) (7440-36-0)(7440-02-0)DIOXIN Fotal

Dissolved Metals 3030E/3120B for PAGE V-3 analyzed using procedure Note: All metals were EPA Form 3510-2C (8-90)

CONTINUE ON REVERSE

from Standard Methods (18)

CONTINUED FROM THE FRONT

| CONTINUED TROM THE TRON                          | NOVE DEL                          |               |                    |                               |  |                               |          | ,          | 0       | T/1 4 Tri 1             |                      |          |
|--|-----------------------------------|---------------|--------------------|-------------------------------|--|-------------------------------|----------|------------|---------|-------------------------|----------------------|----------|
| 1 POLLITANT                                      | Z. N                              | Z. MARK X     |                    |                               | S. EFFLUENI                            |                               |          | 4. UNI S   | 0       | 5. INTARE (optional)    | = (optional)         |          |
|  | ë                                 | ρ.            |                    | a. MAXIMUM DAILY VALUE        | b. MAXIMUM 30 DAT VALUE (if available) | VALUE (if available)          | C        | L          |         | AVERAGE VALUE           |                      | L<br>C   |
| (if available)                                   | TESTING BELIEVED REQUIRED PRESENT | ELIEVED BELIE | BELIEVED ABSENT CC | (1)<br>CONCENTRATION (2) MASS | (1)<br>CONCENTRATION (2) MASS          | (1)<br>CONCENTRATION (2) MASS | ANALYSES | a. CONCENT | b. MASS | (1)<br>CONCENTRATION (2 | (2) MASS AN          | ANALYSES |
| GC/MS FRACTION - VOLATILE COMPOUNDS              | - VOLATILE                        | COMPOUNDS     |                    |                               |  |                               |          |            |         |                         |                      |          |
| 1V. Accrolein<br>(107-02-8)                      |                                   | ^             | ×                  |                               |  |                               |          |            |         |                         |                      |          |
| 2V. Acrylonitrile<br>(107-13-1)                  |                                   |               | X                  |                               | n.                                     |                               |          |            |         |                         |                      |          |
| 3V. Benzene ★ (71-43-2)                          | ×                                 |               |                    | <0.10                         |  |                               | -        | hg/L       |         |                         |                      |          |
| 4V. Bis (Chloro-<br>methyr) Ether<br>(542-88-1)  |                                   | *             | ×                  |                               |  |                               |          |            |         |                         |                      |          |
| 5V. Bromoform<br>(75-25-2)                       |                                   |               | X                  |                               |  |                               |          |            |         |                         |                      |          |
| 6V. Carbon<br>Tetrachloride<br>(56-23-5)         |                                   |               | ×                  |                               |  |                               |          |            |         |                         |                      |          |
| 7V. Chlorobenzene<br>(108-90-7)                  |                                   | ^             | X                  |                               |  |                               |          |            |         |                         |                      |          |
| 8V. Chlorodi-<br>bromomethane<br>(124-48-1)      |                                   | ^             | ×                  |                               |  | 8                             |          |            |         |                         |                      |          |
| 9V. Chloroethane<br>(75-00-3)                    |                                   | ×             | ×                  |                               |  |                               |          |            |         |                         |                      |          |
| 10V. 2-Chloro-<br>ethylvinyl Ether<br>(110-75-8) |                                   | . *           | ×                  |                               |  |                               |          |            |         |                         |                      |          |
| 11V. Chloroform<br>(67-66-3)                     |                                   |               | X                  |                               |  |                               |          |            |         |                         |                      |          |
| 12V. Dichloro-<br>bromomethane<br>(75-27-4)      |                                   |               | X                  |                               |  |                               |          |            |         |                         |                      |          |
| 13V. Dichloro-<br>difluoromethane<br>(75-71-8)   |                                   | *             | ×                  |                               |  |                               |          |            |         |                         |                      |          |
| 14V, 1,1-Dichloro-<br>ethane (75-34-3)           |                                   |               | X                  |                               |  | _                             |          |            |         |                         |                      |          |
| 15V. 1.2-Dichloro-<br>ethane (107-06-2)          |                                   |               | X                  |                               |  |                               |          |            |         |                         |                      |          |
| 16V. 1,1-Dichloro-<br>ethylene (75-35-4)         |                                   | ^             | X                  |                               | 12                                     |                               |          |            |         |                         |                      |          |
| 17V. 1,2-Dichloro-<br>propane (78-87-5)          |                                   |               | ×                  |                               |  |                               |          |            |         |                         |                      |          |
| 18V. 1,3-Dichloro-<br>propylene<br>(542-75-6)    |                                   |               | X                  |                               |  |                               |          | æ          |         |                         |                      |          |
| 19V. Ethylbenzene<br>(100-41-4)                  |                                   | ^             | ×                  |                               |  |                               |          |            |         |                         |                      |          |
| 20V. Methyl<br>Bromide (74-83-9)                 |                                   | ^             | ×                  |                               |  |                               |          |            |         |                         |                      |          |
| 21V. Methyl<br>Chloride (74-87-3)                | 78                                | *             | ×                  |                               |  |                               |          |            |         |                         |                      |          |
| EPA Form 3510-2C (8-90)                          | (8-90)                            |               |                    |                               | PAGE                                   | PAGE V-4                      |          |            |         | CONT                    | CONTINUE ON PAGE V-5 | GE V-5   |

EPAForm 3510-2C(8-90) \* - Sample was analyzed for Methoxychlor (aka Benzene) CAS# 72-43-5.

CONTINUED FROM PAGE V-4

| TOTAL DEPOSIT NOO                                 | >                   | 2 MARK "X"          |           |                          | 3 FEFI LIENT |                         |                   | 4. UNITS     | ITS     | 5. INTAKE (optional)          | (al)                  |
|---|---------------------|---------------------|-----------|--------------------------|--------------|-------------------------|-------------------|--------------|---------|-------------------------------|-----------------------|
| 1. POLLUTANT<br>AND                               |                     | ٤                   |           | E MAXIMIM MINIMIX VI I I | b. MAXIMUM   | c. LONG TERM AVRG.      |                   |              |         | a. LONG TERM<br>AVERAGE VALUE |                       |
|   | TESTING<br>REQUIRED | BELIEVED<br>PRESENT | BELIEVED  |                          | CONCENT      | (1) CONCENTRATION (2) M | (2) MASS ANALYSES | F a. CONCEN- | b. MASS | (1)<br>CONCENTRATION (2) MASS | D. NO. OF<br>ANALYSES |
| GC/MS FRACTION - VOLATILE COMPOUNDS (continued)   | - VOLATIL           | E COMPOL            | UNDS (com |                          | 5            |                         |                   |              |         |                               |                       |
| 22V. Methylene<br>Chloride (75-09-2)              |                     |                     | ×         |                          |              |                         |                   |              |         |                               |                       |
| 23V. 1,1,2,2-<br>Tetrachloroethane<br>(79-34-5)   |                     |                     | X         |                          |              |                         |                   | -            |         |                               |                       |
| 24V. Tetrachloro-<br>ethylene (127-18-4)          |                     |                     | ×         |                          |              |                         |                   |              |         |                               |                       |
| 25V. Toluene<br>(108-88-3)                        |                     |                     | ×         |                          |              |                         |                   |              |         |                               |                       |
| 26V. 1,2-Trans-<br>Dichloroethylene<br>(156-60-5) |                     |                     | X         |                          |              |                         |                   |              |         |                               |                       |
| 27V. 1,1,1-Trichloro-<br>ethane (71-55-6)         |                     |                     | ×         |                          |              |                         |                   |              |         |                               |                       |
| 28V. 1,1,2-Trichloro-<br>ethane (79-00-5)         |                     |                     | ×         |                          |              |                         |                   |              |         |                               |                       |
| 29V Trichloro-<br>ethylene (79-01-6)              |                     |                     | ×         |                          |              |                         |                   |              |         |                               |                       |
| 30V. Trichloro-<br>fluoromethane<br>(75-69-4)     |                     |                     | ×         |                          |              |                         |                   |              |         |                               |                       |
| 31V. Vinyl Chloride<br>(75-01-4)                  |                     |                     | ×         |                          |              |                         |                   |              |         |                               |                       |
| GC/MS FRACTION - ACID COMPOUNDS                   | - ACID CO           | MPOUNDS             | (0        |                          |              |                         |                   |              | -       |                               |                       |
| 1A, 2-Chlorophenol<br>(95-57-8)                   |                     |                     | ×         |                          |              |                         |                   |              |         |                               |                       |
| 2A, 2,4-Dichloro-<br>phenol (120-83-2)            | ×                   |                     |           | <10                      |              |                         | Н                 | hg/L         |         |                               |                       |
| 3A, 2,4-Dimethyl-<br>phenol (105-67-9)            |                     |                     | X         |                          |              |                         |                   |              |         |                               |                       |
| 4A, 4,6-Dinitro-O-<br>Cresol (534-52-1)           | X                   |                     |           | <10                      |              |                         | П                 | ng/L         |         |                               |                       |
| 5A. 2,4-Dinitro-<br>phenol (51-28-5)              |                     |                     | ×         |                          |              |                         |                   |              |         |                               |                       |
| 6A, 2-Nitrophenol<br>(88-75-5)                    |                     |                     | ×         |                          |              |                         |                   |              |         |                               |                       |
| 7A. 4-Nitrophenol (100-02-7)                      |                     |                     | ×         |                          |              |                         |                   |              |         |                               |                       |
| 8A, P-Chloro-M-<br>Cresol (59-50-7)               |                     |                     | ×         |                          |              |                         |                   |              |         |                               |                       |
| 9A. Pentachloro-<br>phenol (87-86-5)              |                     |                     | ×         |                          |              |                         |                   |              |         |                               |                       |
| 10A. Phenol<br>(108-95-2)                         |                     |                     | ×         |                          |              |                         |                   |              |         |                               |                       |
| 11A, 2,4,6-Trichloro-<br>phenol (88-05-2)         |                     |                     | ×         |                          |              |                         |                   |              |         |                               |                       |
| EPA Form 3510-2C (8-90)                           | (8-90)              |                     |           |                          | PAG          | PAGE V-5                |                   |              |         | CONTINUE ON REVERSE           | N REVERSE             |

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|---|-----------|---------------------|----------|-------------------------------|-------------------------------|----------------------|------------|----------|---------|---------|------------------------|----------------------|
| 1. POLLUTANT  | 7         | Y YYYYY             |          |                               | b. MAXIMUM 30 DAY VALUE       | C. LONG TERM         | AVRG.      |          | 4.      | 0       | a. LONG TERM           | nona)                |
|   | a.        | ن                   | ű        | a. MAXIMUM DAILY VALUE        | (if available)                | VALUE (if available) |            | CI       | CONCE   |         | AVERAGE VALUE          |                      |
|   | REQUIRED  | BELIEVED<br>PRESENT | BELIEVED | (1)<br>CONCENTRATION (2) MASS | (1)<br>CONCENTRATION (2) MASS | (1)<br>CONCENTRATION | (2) MASS A | ANALYSES | TRATION | b. MASS | CONCENTRATION (2) MASS | ASS ANALYSES         |
| GC/MS FRACTION - BASE/NEUTRAL COMPOUNDS               | - BASE/NE | EUTRAL CO           | MPOUNDS  | S                             |                               |                      |            |          |         |         |                        |                      |
| 1B. Acenaphthene (83-32-9)                            |           |                     | $\times$ |                               |                               |                      |            |          |         |         |                        |                      |
| 2B. Acenaphtylene (208-96-8)                          |           |                     | ×        |                               |                               |                      |            |          |         |         |                        |                      |
| 3B. Anthracene<br>(120-12-7)                          |           |                     | X        |                               |                               |                      |            |          |         |         |                        |                      |
| 4B. Benzidine<br>(92-87-5)                            | ×         |                     |          | <10                           |                               |                      |            | н        | ng/L    |         |                        |                      |
| 5B. Benzo (a)<br>Anthracene<br>(56-55-3)              |           |                     | X        |                               |                               |                      | 1          |          |         |         |                        |                      |
| 6B. Benzo ( <i>a</i> )<br>Pyrene (50-32-8)            |           |                     | ×        |                               |                               |                      |            |          |         |         |                        |                      |
| 7B. 3,4-Benzo-<br>fluoranthene<br>(205-99-2)          |           |                     | X        |                               |                               |                      |            |          |         |         |                        |                      |
| 8B. Benzo (ghi)<br>Perylene (191-24-2)                |           |                     | ×        |                               |                               |                      |            |          |         |         |                        |                      |
| 9B. Benzo (k)<br>Fluoranthene<br>(207-08-9)           |           |                     | X        |                               |                               |                      |            |          |         |         |                        |                      |
| 10B, Bis (2-Chloro-<br>ethoxy) Methane<br>(111-91-1)  |           |                     | ×        |                               |                               |                      |            |          |         |         |                        |                      |
| 11B. Bis (2-Chloro-<br>ethyl) Ether<br>(111-44-4)     | ×         |                     |          | <10                           |                               |                      |            | H        | ng/L    |         |                        |                      |
| 12B, Bis (2-<br>(*Morotsopropyf)<br>Ether (102-80-1)  | ×         |                     |          | <10                           |                               |                      |            | П        | ng/L    |         |                        |                      |
| 13B. Bis (2-l.thv/-<br>hevy/) Phthalate<br>(117-81-7) |           |                     | ×        |                               |                               |                      |            |          |         |         |                        |                      |
| 14B. 4-Bromophenyl<br>Phenyl Ether<br>(101-55-3)      |           |                     | ×        |                               |                               |                      |            |          |         |         |                        |                      |
| 15B. Butyl Benzyl<br>Phthalate (85-68-7)              |           |                     | ×        |                               |                               |                      |            |          |         |         |                        |                      |
| 16B. 2-Chloro-<br>naphthalene<br>(91-58-7)            | ×         |                     |          | <10                           |                               |                      |            | П        | ng/L    |         |                        |                      |
| 17B. 4-Chloro-<br>phenyl Phenyl Ether<br>(7005-72-3)  |           |                     | ×        |                               |                               |                      |            |          |         |         |                        |                      |
| 18B. Chrysene<br>(218-01-9)                           |           |                     | ×        |                               |                               |                      |            |          |         |         |                        |                      |
| 19B. Dibenzo (a.h)<br>Anthracene<br>(53-70-3)         |           |                     | X        |                               |                               |                      |            |          |         |         |                        |                      |
| 20B. 1,2-Dichloro-<br>benzene (95-50-1)               |           |                     | ×        |                               |                               |                      |            |          |         |         |                        |                      |
| 21B. 1,3-Di-chloro-<br>benzene (541-73-1)             |           |                     | ×        |                               |                               |                      |            |          |         |         |                        |                      |
| EPA Form 3510-2C (8-90)                               | (8-90)    |                     |          |                               | PAGE                          | PAGE V-6             |            |          |         |         | CONTINU                | CONTINUE ON PAGE V-7 |

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| TOON INCIDENT TAGE                                    | >                                 | ANADAM C  |          |                               | 3 EEEL LENT                            |                        |          | STINIT  | TC      | 5 INTAKE (optional)    | Vio       |
|---|-----------------------------------|-----------|----------|-------------------------------|--|------------------------|----------|---------|---------|------------------------|-----------|
| 1. POLLUTANT  | 7                                 | MARK Y    |          |                               | 5. EFFLUEINI<br>B MAXIMIM 30 DAY VALUE |                        |          | 5       | 2       | a LONG TERM            | an)       |
| AND   | cij                               | Ď.        | ú        | a, MAXIMUM DAILY VALUE        | (if available)                         | VALUE (if available)   | 2        | N C C   |         | AVERAGE VALUE          | 2         |
| (if available)  | TESTING BELIEVED REQUIRED PRESENT | PRESENT   | BELIEVED | (1)<br>CONCENTRATION (2) MASS | CONCENTRATION (2) MASS                 | CONCENTRATION (2) MASS | ANALYSES | TRATION | b. MASS | CONCENTRATION (2) MASS | ANALYSES  |
| GC/MS FRACTION - BASE/NEUTRAL COMPOUNDS (continued)   | 1 - BASE/NE                       | EUTRAL CO | OMPOUND  | S (continued)                 |  |                        |          |         |         |                        |           |
| 22B. 1,4-Dichloro-<br>benzene (106-46-7)              |                                   |           | X        |                               |  |                        |          |         |         |                        |           |
| 23B. 3,3-Dichloro-<br>benzidine (91-94-1)             | ×                                 |           |          | <20                           |  |                        | П        | ng/L    |         |                        |           |
| 24B. Diethyl<br>Phthalate (84-66-2)                   |                                   |           | ×        |                               |  |                        |          |         |         |                        |           |
| 25B. Dimethyl<br>Phthalate<br>(131-11-3)              | ×                                 |           |          | <10                           |  |                        | Т        | ng/L    |         |                        |           |
| 26B. Di-N-Butyl<br>Phthalate (84-74-2)                |                                   |           | X        |                               |  |                        |          |         |         |                        |           |
| 27B. 2,4-Dinitro-<br>toluene (121-14-2)               |                                   |           | X        |                               |  |                        |          |         |         |                        |           |
| 28B. 2,6-Dinitro-<br>toluene (606-20-2)               |                                   |           | ×        |                               |  |                        |          |         |         |                        |           |
| 29B. Di-N-Octyl<br>Phthalate (117-84-0)               |                                   |           | ×        |                               |  |                        |          |         |         |                        |           |
| 30B. 1,2-Diphenylhydrazine (as Azobenzene) (122-66-7) | ×                                 |           |          | <10                           |  |                        | П        | ng/L    |         |                        |           |
| 31B. Fluoranthene<br>(206-44-0)                       |                                   |           | X        |                               |  |                        |          |         |         |                        |           |
| 32B. Fluorene<br>(86-73-7)                            |                                   |           | ×        |                               |  |                        |          |         |         |                        |           |
| 33B. Hexachloro-<br>benzene (118-74-1)                | ×                                 |           |          | <10                           |  |                        | н        | µg/L    |         |                        |           |
| 34B. Hexachloro-<br>butadiene (87-68-3)               | ×                                 |           |          | <10                           |  |                        | н        | hg/L    |         |                        |           |
| 35B. Hexachloro-<br>cyclopentadiene<br>(77-47-4)      | ×                                 |           |          | <1.0                          |  |                        | п        | ng/L    |         |                        |           |
| 36B Hexachloro-<br>ethane (67-72-1)                   | ×                                 |           |          | <10                           |  |                        | -1       | µg/L    |         |                        |           |
| 37B. Indeno<br>(1,2,3-cd) Pyrene<br>(193-39-5)        | ×                                 |           |          | <20                           |  |                        | Т        | hg/L    |         |                        |           |
| 38B. Isophorone<br>(78-59-1)                          |                                   |           | X        |                               |  |                        |          |         |         |                        |           |
| 39B. Naphthalene (91-20-3)                            |                                   |           | X        |                               |  |                        |          |         |         |                        |           |
| 40B. Nitrobenzene<br>(98-95-3)                        |                                   |           | X        |                               |  |                        |          |         |         |                        |           |
| 41B. N-Nitro-<br>sodimethylamine<br>(62-75-9)         | ×                                 |           |          | <10                           |  |                        | н        | ng/L    |         |                        |           |
| 42B. N-Nitrosodi-<br>N-Propylamine<br>(621-64-7)      | ×                                 |           | 2        | <10                           |  |                        | н        | ng/L    |         |                        |           |
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|---|---------------------------|-----------|----------|------------------------|----------|--|-------------|---|----------|-----------------------|------------|---------|------------------------|----------------------|----------|
| FAAF  | Z. IVI                    | Z. WARK X |          |                        |          | 0.                                     | S. EFFLUENI |   | 000      |                       | 4.0        | 0       | O. INI AND             | (opnonal)            |          |
|   | rs                        | Ď.        | ú        | a. MAXIMUM DAILY VALUE | LY VALUE | b. MAXIMUM 30 DAY VALUE (if available) | DAY VALUE   | c. LONG TERM AVRG. VALUE (if available) |          |                       | i i        |         | AVERAGE VALUE          |                      | L<br>C   |
| (if available)                                      | TESTING BE<br>REQUIRED PR | BELIEVED  | BELIEVED |                        | (2) MASS | (1)<br>CONCENTRATION                   | (2) MASS    | (1)<br>CONCENTRATION                    | (2) MASS | d. NO. OF<br>ANALYSES | a. CONCENT | b. MASS | (1)<br>CONCENTRATION ( | (2) MASS AN          | ANALYSES |
| GC/MS FRACTION - BASE/NEUTRAL COMPOUNDS (continued) | - BASE/NEUT               | TRAL CO   | MPOUNDS  | (continued)            |          |  |             |   |          |                       |            |         |                        |                      |          |
| 43B. N-Nitro-<br>sodiphenylamine<br>(86-30-6)       | ×                         |           |          | <10                    |          |  | 8           |   |          | Н                     | µg/L       |         |                        |                      |          |
| 44B. Phenanthrene (85-01-8)                         |                           |           | ×        |                        |          |  |             |   |          |                       |            |         |                        |                      |          |
| 45B. Pyrene<br>(129-00-0)                           |                           |           | X        |                        |          |  |             |   |          |                       |            |         |                        |                      |          |
| 46B. 1,2,4-Tri-<br>chlorobenzene<br>(120-82-1)      |                           |           | X        |                        |          |  |             |   |          |                       |            |         |                        |                      |          |
| GC/MS FRACTION - PESTICIDES                         | - PESTICIDE               | S         |          |                        |          |  |             |   |          |                       |            |         |                        |                      |          |
| 1P. Aldrin<br>(309-00-2)                            |                           |           | X        |                        |          |  |             |   |          |                       |            |         |                        |                      |          |
| 2P. α-BHC<br>(319-84-6)                             |                           |           | X        |                        |          |  |             |   |          |                       |            |         |                        |                      |          |
| 3P. B-BHC<br>(319-85-7)                             |                           |           | X        |                        |          |  |             |   |          |                       |            |         |                        |                      |          |
| 4P. y-BHC (58-89-9)                                 | ×                         |           |          | <0.50                  |          |  |             |   |          | 1                     | hg/L       |         |                        |                      |          |
| 5P. 8-BHC<br>(319-86-8)                             |                           |           | ×        |                        |          |  |             |   |          |                       |            |         |                        |                      |          |
| 6P. Chlordane<br>(57-74-9)                          |                           |           | X        |                        |          |  |             |   |          |                       |            |         |                        |                      |          |
| 7P. 4,4'-DDT (50-29-3)                              |                           |           | X        |                        |          |  |             |   |          |                       |            |         |                        |                      |          |
| 8P. 4,4-DDE<br>(72-55-9)                            |                           |           | X        |                        |          |  |             |   |          |                       |            |         |                        |                      |          |
| 9P. 4,4'-DDD<br>(72-54-8)                           |                           |           | ×        |                        |          |  |             |   |          |                       |            |         |                        |                      |          |
| 10P. Dieldrin<br>(60-57-1)                          |                           |           | ×        |                        |          |  |             |   |          |                       |            |         |                        |                      |          |
| 11P, α-Enosulfan<br>(115-29-7)                      |                           |           | X        |                        |          |  |             |   |          |                       |            |         |                        |                      |          |
| 12P. ß-Endosulfan (115-29-7)                        |                           |           | ×        |                        |          |  |             |   |          |                       |            |         |                        |                      |          |
| 13P. Endosulfan<br>Sulfate<br>(1031-07-8)           |                           |           | X        |                        |          |  |             |   |          |                       |            |         |                        |                      |          |
| 14P. Endrin<br>(72-20-8)                            |                           |           | X        |                        |          |  | M.          |   |          |                       |            |         |                        |                      |          |
| 15P, Endrin<br>Aldehyde<br>(7421-93-4)              |                           |           | ×        |                        |          |  |             |   |          |                       |            |         |                        |                      |          |
| 16P. Heptachlor<br>(76-44-8)                        |                           |           | X        |                        |          |  |             |   |          |                       |            |         |                        |                      |          |
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# OVERVIEW AND DESCRIPTION OF ILUKA RESOURCES INC. MINERAL SEPARATION PLANT AND PROCESS WATER SYSTEM

#### **1.0 OVERVIEW OF OPERATIONS AND WASTEWATER SOURCES**

Iluka Resources' Processing site is situated on 36.6 acres in Sussex County near the town of Stony Creek (see Figure 1). The site contains Iluka's Administration / Laboratory building for the Virginia Operations, as well as the Mineral Separation Plant (MSP), the Effluent Treatment Plant (ETP), and associated storm water management system (see Figure 2 for site layout). Major operations involved in the processing of mineral sands are conducted in and around the MSP and include the following:

- Mineral Sand Concentrate Storage and Plant Feed
- Drying of Concentrate Feed
- Ilmenite and HiTi Circuits
- Zircon Recovery Circuit
- Zircon Finishing Plant Circuit (ZFP)
- Staurolite Removal Circuit
- ETP
- Tailings Removal

A process flow diagram that shows major steps from the mine site to the production of finished products at the MSP is included as Figure 3. The MSP typically operates on a continuous 24 hours per day, 365 days per year schedule. The site currently maintains two outfalls under VPDES Permit No. VA0090981. Outfall 001 serves to discharge treated waste water from the ETP, while outfall 002 discharges storm water from Retention Pond 3\*. Iluka is proposing the addition of outfall 004<sup>†</sup> to serve as a discharge point for process water from Process Water Pond 2.

<sup>\*</sup> Removal of the berm separating Retention Pond 3 from the Sediment Pond (VPDES Permit No. VAR051217) has been proposed to DEQ and is currently being reviewed by the agency.

<sup>&</sup>lt;sup>†</sup> The addition of the Admin Storm water pond's outfall (currently under VPDES Permit No. VAR051217) to Permit #VA0090981 as outfall 003 has been proposed to DEQ and is currently being reviewed by the agency.

| Iluka Resources Inc. – VA Operations



#### 1.1 Mineral Sand Concentrate Storage and Plant Feed

At the Virginia Operations' mines and Mine Concentrator sites, mineral sands ore is excavated from relic beach ridge deposits by track-hoe excavators, slurried, and concentrated using various wet gravity separation processes. Approximately 10 percent by weight of the ore body is removed from the excavated material as mineral sand concentrate while the remaining 90 percent clay and sand tailings are returned to the tailings pit in the mine area, graded, and then reclaimed in accordance with all applicable permits. The mineral sand concentrate is pumped from the Mine Concentrator building to a concentrate stacker where excess water is removed and returned to the Mine Concentrator process water pond. The raw mineral sand concentrate, which consists of approximately 90 percent mineral sands, is then hauled by truck approximately 14 miles to the MSP for final separation into the ilmenite, zircon, and staurolite mineral sands products. After weigh-in on the scales at the entrance to the MSP, the trucks dump the raw concentrate in an approximately 1.85-acre storage stockpile area located immediately north of the MSP. Raw concentrate is removed from the stockpile area and fed into the MSP's grizzly feed sump by the site's front end loader.

#### 1.2 Drying of Concentrate Feed

Drying is required to reduce moisture content to less than 0.1 percent by weight prior to electrostatic and electromagnetic processing for separation of titanium bearing-minerals (ilmenite, rutile and leucoxene), zircon, staurolite, and non-conducting gangue minerals. The mineral sand concentrate loaded into the grizzly feed sump is slurried with clean water from the Process Water Ponds, screened, and pumped into the 1,600-ton new feed day tank. Mineral slurry from the day tank is pumped to the scrubber rinse circuit and then to the filter feed cyclones to remove excess water prior to additional dewatering on a belt filter. The belt reduces the moisture content of the feed to approximately 5 percent by weight. The filtrate liquid is pumped to the process water system for reuse. The filter cake



from the belt press is temporarily stored in a bin and then screw fed into a fluid-bed dryer to reduce moisture content to less than 0.1 percent by weight.

#### 1.3 Ilmenite and HiTi Circuit

After the concentrate is sufficiently dried it is screened to remove oversize material, which is sent to the tailings stockpile. The underflow from the screen is fed into high-tension electrostatic (HT) separators, which segregate the electrically conductive ilmenite and rutile from the non-conductive zircon, staurolite and gangue minerals. The conductive minerals are screened and the oversize material is slurried and pumped to the tailings stockpile. The material passing through the screen is sent through a series of magnets to separate the more magnetic titanium-bearing mineral ilmenite from the less magnetic titanium-bearing minerals rutile and leucoxene, collectively referred to as HiTi. The streams of magnetic titanium-bearing minerals from each magnet are combined and conveyed to the ilmenite product storage bin prior to shipment. The less magnetic HiTi minerals pass through a series of electrostatic plate separators to remove any residual non-conductive material before being combined with the ilmenite product stream prior to storage in the ilmenite product storage bin. Various properties of the minerals processed at the MSP can be found in Table 1 below.

Table 1: Properties of Minerals

|                     | Ilmenite              | Rutile                    | Leucoxene                             | Zircon                        |
|---------------------|-----------------------|---------------------------|---------------------------------------|-------------------------------|
| Chemical Formula    | FeTiO₃,               | TiO <sub>2</sub>          | TiO <sub>2</sub> + FeTiO <sub>3</sub> | ZrSiO <sub>4</sub>            |
| Specific Gravity    | 4.25                  | 4.22                      | 3.95                                  | 4.69                          |
| Magnetic Properties | Conductor<br>Magnetic | Conductor<br>Non-Magnetic | Conductor<br>Weakly Magnetic          | Non-Conductor<br>Non-Magnetic |

## 1.4 Zircon Recovery and Finishing Plant Circuits

Zircon recovery from the non-conductive mineral stream is improved by the use of wet gravity separation methods in the portion of the MSP known as the Wet Section. Here the non-conductive minerals are again slurried using process water and passed through a series of nested spirals and over two shaker tables. These methods exploit the variations in the



specific gravities the minerals to separate the heavier zircon from the other, lighter nonconductive minerals. The zircon sand is then pumped in slurry form to the Zircon Finishing Plant (ZFP). Here, the zircon is bathed in a solution of sulfuric acid to remove iron oxides coatings from the surfaces of the grains. The acidic solution is washed from the grains in Rinsing Hydrosizer No. 2 using clean process water from Process Water Pond 2. Treated effluent water from the Effluent Treatment Plant (ETP) is used in all other rinsing steps in the ZFP. The overflow from Rinsing Hydrosizer No. 2 empties into the Rinsing Sump where it is pumped to the ETP for neutralization and treatment. The cleaned zircon concentrate underflow from the Hydrosizer is discharged into a sump and then pumped into a final dewatering cyclone. The dewatered zircon concentrate is conveyed to a vacuum filter belt for additional dewatering and then to the zircon fluid bed dryer for removal of any remaining moisture. The dried zircon concentrate is screened to remove oversized material and fed into a secondary three-stage HT separator to remove any residual conductive material. This conductive material is returned to the primary HT circuit to recover any titanium-bearing minerals. The non-conductive material is passed through a high-intensity magnetic separator circuit to remove any residual magnetic material. The magnetic material is sent to the tailings stockpile while the non-magnetic material (primarily zircon) is screened and conveyed to the zircon product storage bins for shipment.

#### 1.5 Staurolite Removal Circuit

The Staurolite Removal Circuit is also contained within the MSP. The non-conductor stream from the Ilmentite and HiTi Circuit is combined with the non-conductor product from the initial electrostatic separation process to form the feed for the Staurolite Removal Circuit. A bucket elevator and conveyor transport the feed to a set of rare earth roll magnetic separators (RERMS), which produce separate mag1, mag2 and non-mag material streams. The top roll, with its lower magnetic rating, recovers highly magnetic material (mag1); primarily misplaced ilmenite. The mag1 material is directed to the base of the elevator that feeds the conductor screens. The second and third rolls recover paramagnetic material



(mag2) that is passed through an electrostatic separator to scavenge any misplaced conductor material. The paramagnetic conductor stream is sent to the conductor screen elevator along with the mag1 product. The paramagnetic non-conducting staurolite-containing material is presently sent to the conductor oversize sump and from there to the staurolite stacker for disposal as tailings. However, thanks to recent modifications, the staurolite stream can now be diverted to the HiTi storage bin to await shipping if a market for it is discovered. The non-magnetic stream from the RERMS is be directed to a surge bin for storage or as feed to the Zircon Recovery Circuit.

#### 1.6 Tailings

Tailings are slurried and pumped to the tailings stacker located immediately west of the MSP. Excess water from the tailings stacker drains into the Stacker (Process) Turkey Nest. An electric-powered pump then transfers water from the Turkey Nest to the Process Water Ponds for reuse. The dewatered and stacked tailings are loaded into dump trucks and hauled back to the mine sites where they are backfilled into the mine pits and graded for reclamation.

#### 2.0 WATER QUALITY

#### 2.1 Process Water Quality, Storage, Treatment, and Proposed Discharge

The mineral sand concentrate processed at the MSP includes the titanium-bearing minerals ilmenite, rutile, and leucoxene, in addition to zircon, staurolite and miscellaneous gangue minerals. By taking advantage of their differing physical properties (see Table 1), these minerals are separated from each other using non-chemical techniques which include various wet gravity methods, high-tension electrostatic separation, and magnetic separation. The overall quality of the process water is not expected to be effected by the minerals handled in the MSP due to their relatively insoluble nature, and the lack of biological waste components. Process water is simply used to move the products in slurry



form or to facilitate gravity separation (Please see Figure 4 for a water balance diagram of the Process system). Process water is stored in two ponds designated Process Ponds 1 and 2, respectively. In these ponds the water is treated by sedimentation and, to a lesser extent aeration. The pH of the process water declines slowly over time due to the influence of the Primary and Zircon Dryer Stack Scrubbers (VA DEQ Air Permit Reference No. 51220) and so periodic additions of sodium hydroxide (Na<sub>2</sub>OH) or hydrated lime are needed to maintain the level within the optimum range of 6 to 9 SU. The pH buffer is typically introduced at the Stacker Turkey nest to allow for maximum mixing and dissolution. Environment, Health & Safety as well as MSP personnel monitor the pH level of the Process Ponds on a regular basis using field pH meters that are standardized annually by the manufacturer (YSI) (see Attachment 1) or an authorized 3<sup>rd</sup> party, and calibrated according to USGS method I-1586-85. Monitoring is conducted in accordance with techniques established in the 1982 edition of "Techniques of Water-Resources Investigations of the United States Geological Survey, Chapter A6, Quality Assurance Practices for the Chemical and Biological Analyses of Water and Fluvial Sediments."

Iluka proposes to discharge treated Process water from Process Water Pond 2. This pond is equipped with a concrete spillway that was installed during its construction to protect the impoundment from breaching if overtopped due to severe rain events. The proposed discharge would flow down the concrete spillway and enter a culvert to be installed underneath the site access road on the east side of the pond. From there the water will pass through an existing culvert to an onsite ditch, both of which are currently used to convey storm water from the site's Sediment Pond<sup>‡</sup> (VPDES #VAR051217). The flow will then exit the site in the same location as outfalls 001 and 002.

+

<sup>&</sup>lt;sup>‡</sup> Sealing of this pond's outfall and combining its volume with Retention Pond 3 to discharge through outfall 002 has been proposed to DEQ.

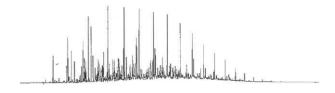


### 2.1.1 Analytical Testing

Lab reports resulting from analytical testing of the process water are included with this application. All analyses were conducted by Primary Laboratories in Mechanicsville, Virginia.

## Primary Laboratories, Inc.

7423 Lee Davis Road • Mechanicsville, VA 23111 • Telephone (804) 559-9004 • Fax (804) 559-9306



### ANALYTICAL LABORATORY REPORT

7-Mar-08

ILUKA Resource, Inc. Attn: Kevin Rideout

12472 St. John Church Road Stony Creek, Virginia 23882

Date Received:

4-Mar-08

Date Sampled:

3-Mar-08

Work Order No:

0803005-01

Client ID:

MSP Process Pond

| Test        | Final  | Reporting | Units of | Standard     | Date     | Time     | Tech.    |
|-------------|--------|-----------|----------|--------------|----------|----------|----------|
| Description | Result | Limit     | Measure  | Methods (18) | Analyzed | Analyzed | Initials |
| TSS         | 11.5   | 1.0       | mg/L     | 2540 D       | 6-Mar-08 | 11:00    | AS       |

Note: Sample received in laboratory on ice.

Signature:

Parely L. Bragg

Laboratory Manager

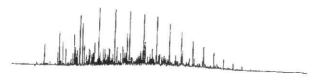
Date: 3/4/08

These analytical results are based upon materials provided by the client and are intended for the exclusive use of the client. These analytical results represent the best judgement of Primary Laboratories, Inc. Primary Laboratories, Inc. assumes no responsibility, express or implied, as to the interpretation of the analytical results contained in this report. This report is not to be reproduced except with the written approval of Primary Laboratories, Inc.

## Primary Laboratories, Inc.

8045599306

7423 Lee Davis Road • Mechanicsville, VA 23111 • Telephone (804) 559-9004 • Fax (804) 559-9306



# ANALYTICAL LABORATORY REPORT

16-Apr-08

Iluka Resources, Inc. Attn: Kevin Rideout

12472 St Johns Church Road Stoney Creek, VA 23882

Date Received:

26-Feb-08

Date Sampled:

26-Feb-08 0802205-01

Work Order No:

| Client ID:       | MSP Process | Danda     |           |                      |                                   |          |
|------------------|-------------|-----------|-----------|----------------------|-----------------------------------|----------|
| Test             | Final       |           | T 11 ===  |                      |                                   |          |
| Description      | Result      | Reporting |           | Standard             | Date                              | Tech     |
| Dissolved Metals | Rosult      | Limit     | Measure   | Methods (18)         | Analyzed                          | Initials |
| Copper           | <0.020      | 0.020     | mg/L      | 3030E/3120B          | 28-Feb-08                         | HV       |
| Iron             | <0.050      | 0.050     | mg/L      | 3030E/3120B          | at 12:15<br>29-Feb-08             | HV       |
| Lead             | <0.050      | 0.050     | mg/L      | 3030E/3120B          | at 14:12<br>28-Feb-08             | HV       |
| Nickel           | <0.020      | 0.020     | mg/L      | 3030E/3120B          | at 12:15<br>28-Feb-08             | HV       |
| Selenium         | <0.050      | 0.050     | mg/L      | 3030E/3120B          | at 12:15<br>28-Feb-08             | HV       |
| Thallium         | <0.002      | 0.002     | mg/L      | 3030E/3113B          | at 12:15<br>28-Feb-08             | HV       |
| Titanium         | <0.020      | 0.020     | mg/L      | 3030E/3120B          | at 9:30<br>29-Feb-08              | HV       |
| Zinc             | 0.020       | 0.010     | mg/L      | 3030E/3120B          | at 14:12<br>28-Feb-08<br>at 12:15 | HV       |
| Hydrogen Sulfide | <0.05       | 0.05      | mg/L      | 4500S <sub>2</sub> E | 28-Feb-08                         | HV       |
| E. coli          | <2          | 2         | MPN/100ml | 9221 F               | at 11:00<br>26-Feb-08             | AS       |
| Chlorides        | 24.1        | 0.1       | mg/L      | 4500CL B             | at 16:45<br>28-Feb-08             | NA       |
| oc               | 3.31        | 1.00      | mg/L      | 5310 C               | at 14:30<br>28-Feb-08             | PB       |
| OD               | 30.5        | 4.0       | mg/L      | 5520 C               | at 13:00<br>28-Feb-08             | NA       |

### Primary Laboratories, Inc. Results

16-Apr-08

Date Sampled:

26-Feb-08

Work Order No:

0802205-01

Client ID:

MSP Process Ponds

| CHETTE ID.           | Mar Process I   | onas               |                     |                          |                                |       |
|----------------------|-----------------|--------------------|---------------------|--------------------------|--------------------------------|-------|
| Test<br>Description  | Final<br>Result | Reporting<br>Limit | Units of<br>Measure | Standard<br>Methods (18) | Date<br>Analyzed               | Tech. |
| BOD                  | <3.0            | 3.0                | mg/L                | 5210 B                   | 27-Mar-08                      | NA    |
| Ammonia              | 1.33            | 0.01               | mg/L                | 4500NH <sub>3</sub> F    | at 12:00<br>28-Feb-08          | NA    |
| Strontium-90         | 0.4 +/- 0.3     | 0.5                | pCi/L               | 905.0                    | at 8:00<br>26-Mar-08           | sc*   |
| Tritium              | 4.8 +/- 87.5    | 146.9              | pCi/L               | 906.0                    | 11-Apr-08                      | SC*   |
| Beta Particle Photon | 13.2 +/- 1.0    | 1.5                | pCi/L               | 900.0                    | 5-Mar-08                       | SC*   |
| Gross Alpha Particle | 17.8 +/- 2.3    | 2.0                | pCi/L               | 900.0                    | at 8:00<br>5-Mar-08<br>at 8:00 | SC*   |

<sup>\*</sup> Analysis sub-contracted.

Units of Measure:

ug/L

Standard Methods (18):

608

Date Analyzed: Time Analyzed:

28-Feb-08 12:28

Technician:

HV

Date Sampled:

26-Feb-08

Work Order No:

0802205-01

| Client ID:          | MSP Process Ponds |                    |          |  |  |  |
|---------------------|-------------------|--------------------|----------|--|--|--|
| Test<br>Description | Final<br>Result   | Reporting<br>Limit | CAS#     |  |  |  |
| Pesticides          |                   |                    |          |  |  |  |
| Gamma-BHC (Lindane  | < 0.50            | 0.50               | 58-89-9  |  |  |  |
| Methoxychlor        | < 0.10            | 0.10               | 72-43-5  |  |  |  |
| Hexachlorobenzene   | < 0.10            | 0.10               | 118-74-1 |  |  |  |
| PCB 1016            | <1.0              | 1.0                |          |  |  |  |
| PCB 1221            | <1.0              | 1.0                |          |  |  |  |
| PCB 1232            | <1.0              | 1.0                |          |  |  |  |
| PCB 1242            | <1.0              | 1.0                |          |  |  |  |
| PCB 1248            | <1.0              | 1.0                |          |  |  |  |
| PCB 1254            | <1.0              | 1.0                |          |  |  |  |
| PCB 1260            | <1.0              | 1.0                |          |  |  |  |

# Primary Laboratories, Inc. Results

16-Apr-08

Standard Methods (18):

625

Date Analyzed:

28-Feb-08

Time Analyzed:

10:54

Technician:

HV

Date Sampled:

26-Feb-08

Units of Measure:

ug/L

Work Order No:

0802205-01

Client ID:

MSP Process Ponds

| Ollette ID.                   | MSP Process Ponds |           |          |  |  |
|-------------------------------|-------------------|-----------|----------|--|--|
| Test                          | Final             | Detection |          |  |  |
| Description                   | Result            | Limit     | CAS#     |  |  |
| Benzidine                     | <10               | 10        | 92-87-5  |  |  |
| bis-(2-Chloroethyl)ether      | <10               | 10        | 111-44-4 |  |  |
| bis-(2-Chloroisopropyl )ether | <10               | 10        | 108-60-1 |  |  |
| 2-Chloronaphthalene           | <10               | 10        | 91-58-7  |  |  |
| 3,3-Dichlorobenzidine         | <20               | 20        | 91-94-1  |  |  |
| Dimethyl phthalate            | <10               | 10        | 131-11-3 |  |  |
| 2,4-Dinitrophenol             | <10               | 10        | 120-83-2 |  |  |
| 2-Methyl-4,6-Dinitrophenol    | <10               | 10        | 534-52-1 |  |  |
| Hexachlorobutadiene           | <10               | 10        | 87-68-3  |  |  |
| Hexachlorocyclopentadiene     | <10               | 10        | 77-47-4  |  |  |
| Hexachloroethane              | <10               | 10        | 67-72-1  |  |  |
| Indeno(1,2,3-cd) pyrene       | <20               | 20        | 193-39-5 |  |  |
| N-Nitrosodimethylamine        | <10               | 10        | 62-75-9  |  |  |
| N-Nitrosodiphenylamine        | <10               | 10        | 86-30-6  |  |  |
| N-Nitrosodi-n-propylamine     | <10               | 10        | 621-64-7 |  |  |
| 1,2-Diphenylhydrazine         | <10               | 10        | 122-66-7 |  |  |
|                               |                   |           |          |  |  |

Note: Samples received in laboratory on ice.

Signature

Parry L. Braco

Laboratory Manager

Date.\_\_\_\_\_

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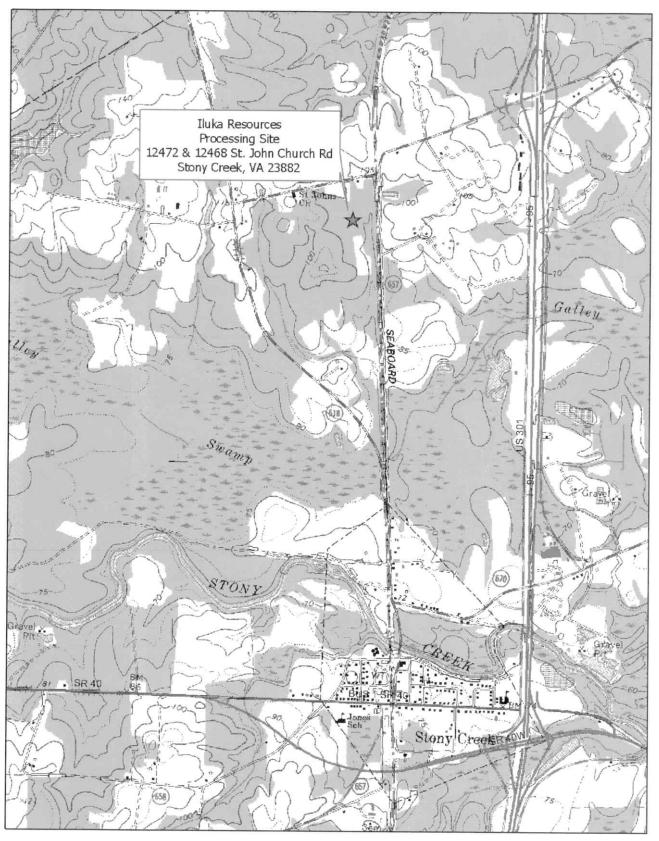


Figure 1. Iluka Resources Processing Site Location Map

Miles

